University of Stavanger

Faculty of Science and Technology

BACHELOR`S THESIS

Study program/Specialization: Petroleum Technology	Spring semester, 2021 Open
Writer: Christian Høie	............Christian Høie.................. (Writer's signature)
Faculty supervisor: Hans Joakim Skadsem	
Thesis title: Permeability of Cement Plugs In Steel Pipes and Effects of Treatment Material Injection	
Credits (ECTS): 20	
Key words: Aperture Microannuli ThermaSet Resin Remedial Treatment	Pages: 53 Stavanger, 26th of June 2021

Abstract

Wellbore integrity is a common challenge in the oil and gas industry. Despite all measures in place, the integrity is often threatened. The integrity of the well is dependent on zonal isolation, effort is made to ensure that there is no fluid migration possible. However, sustained casing pressure is a common problem in the industry.

Poor primary cement jobs are often the culprit of well integrity issues. A poor primary cement job requires additional cementing work, remedial cementing. The traditional method of remedial cementing is squeeze cementing, often using ordinary Portland cement. However, success rates are often low as the slurry is unable to penetrate the relevant apertures due to the particle size. Alternative remedial materials have thus been developed, such as Wellcem's ThermaSet®.

This thesis studied the permeability of cement plugs cast in steel casings and the effects of treatment material injection in an attempt to reduce the permeability, which was higher than what is considered for bulk cement due to leakage pathways. Five steel casings were filled with API Neat class G cement, the samples were leakage tested to establish the permeability. At the conclusion of the permeability measurements, the samples were subject to lab-scale treatment experiments using two different recipes of ThermaSet. The permeability measurements before and after the treatment was compared. Three out of five samples experienced a reduction in permeability.

Acknowledgements

This thesis would not have been possible without the guidance of Hans Joakim Skadsem. I wish to express my deepest gratitude for the motivation and input you have provided me over this strange and restrictive period of COVID-19. The meetings and conversations have been of great help in how to best complete my bachelor's thesis. Thank you.

I would also like to thank Adijat Ayobami Ogienagbon for your help in the laboratory, your inputs and knowledge of laboratory work is greatly appreciated.

Lastly, I would like to thank Dagfinn Søndenaa Sleveland for your help and knowledge of the equipment used in the laboratory. You have been of great help in the understanding of the equipment used.

Table of Content

Table of Figures

Table of Tables

Table of Equations

1 Introduction

1.1 Background

Wellbore integrity is a common challenge in the oil and gas industry. During the design and drilling of any well, a mutual objective is to keep costs at a minimum and production at a maximum. By applying technical, operational and organizational solutions, the most critical objective is to provide an effective zonal isolation between the producing zones and the surface during the life of a well. Effort is made to ensure that there is no fluid migration possible through the cemented annulus. Despite all the measures in place, sustained casing pressure (SCP) is a common problem in the industry (Ali et al., 2021).

Poor cement jobs are causing the oil and gas industry tremendous costs. If a poor cement job is left unattended, the result can be catastrophic. A poor cement job requires additional cementing work, the remedial cementing is both time consuming and costly. Evidently, there is room for improvement in cementing methods to withstand the rigors of well operations and any disruptions that might occur during the lifecycle.

The traditional process of reestablishing zonal isolation takes use of squeeze cementing (Wigston et al., 2019). In short, communication to the leakage source has to be established in order to inject a sealant to remediate the leak. This is traditionally done by perforation of the casing and typically also the cement sheath. The most common sealant material is ordinary Portland cement (OPC); however, the success rate of remedial treatment is low, often due to the particle size of the OPC (Nelson & Guillot, 2006). Alternative particle free treatment materials have thus been developed.

1.2 Objective of thesis

The objective of this thesis is to study the permeability of cement plugs cast in steel casings and the effect of treatment material injection to reduce the permeability. To study the effectiveness of treatment, using two different resin formulations, lab-scale treatment experiments were performed and the permeabilities before and after treatment were compared.

1.3 Structure of thesis

The thesis starts with an introduction to the problem of SCP, describing relevant theory along the way, defining SCP and quantifying the issue by looking at some statistics. Moving forward, theory of primary cementing and the problems that arise from annular fluid migration are highlighted. Thus, the theory of remediation strategies and problems with the current strategies can be presented, before sowing the information presented in the thesis toward the experimental part. Five small scale steel casings filled with API Neat class G cement are permeability tested, at the conclusion of the permeability testing the samples are injected with ThermaSet® to reduce the permeability.

2 Sustained Casing Pressure

Sustained casing pressure is defined as any measurable pressure that persistently rebuilds after bleed-down, attributable to causes other than artificially applied pressure or temperature fluctuations in the well imposed by the operator (Rocha-Valadez et al., 2014; Welltec,). Many producing and abandoned wells exhibit this problem, and it may be caused by leakage of gas through channels in the cement or through the cement-steel interface, the microannuli.

SCP is a widespread well integrity issue which affects more than 30% of wells globally and has cost operator over 75 billion USD since 2009 (Welltec,). This has provided the impetus for the industry to focus on and rework the international standards on well integrity management (Welltec,).

Not only is SCP a well integrity issue, but also an environmental and health risk. From risk of groundwater contamination to major blowouts. In the Western Canada the surface casing vents (SCV) must be left open, allowing natural gas to freely escape to the atmosphere. Jurisdictions in the United States dictates that the SCV must be kept closed (Wigston et al., 2019). However, keeping the SCV closed allows pressure buildup in the annulus where the pressure can continue to build until it is greater than the pressure surrounding water aquifers and result in inflow of gas.

3 Quantifying the issues of SCP

To ensure a satisfactory production, the production optimization begins with a good well completion, which is dependent on the integrity of the well. The well integrity is highly dependent on a proper primary cement job. A 1995 study by Westport Technology revealed that about 15% of primary cement jobs fail, costing the upstream oil and gas industry an estimated USD 450 million (would be USD 795 million today taking into account inflation) annually in remedial cementing work (Newman et al., 2001). It was also stated that approximately one third of the problems is attributable to gas migration or formation water flow during placement and curing of the cement. Figure 3.1 shows the percentage of wells experiencing SCP versus the age of the well for 22,000 wells in the U.S. Gulf of Mexico (personal communication, J. Levine, 2003 cited in Nelson & Guillot, 2006). This means that 8,000 to 11,000 wells in the Gulf of Mexico experience SCP.

Watson & Bachu, 2009 revealed through a systematic well-integrity study in Alberta, Canada that 64% of 20,725 wells tested suffered from leakage through surface casing vent (SCV). Poorly cemented casing was cited as a major contributor to the leakage. Vignes & Aadnøy, 2009 studied well-integrity issues of Norway's offshore settings, involving 406 wells. The project indicated that 18% have integrity failure, issues, or uncertainties, and 7% of the wells were shut in due to well-integrity issues. Control of barrier status is an important health, safety, and environmental (HSE) factor to avoid major incidents caused by unintentional leaks and well-control situations.

In addition to producing wells, quantifying the CO_2 leakage rate during sequestration has gained interest as it is a possible emission into the environment. Other possible emissions into the environment are natural gas flow through wellbore leakage, often attributed to the failure of the cement sheath or cement plugs (in abandoned wells) to provide zonal isolation (Wigston et al., 2019).

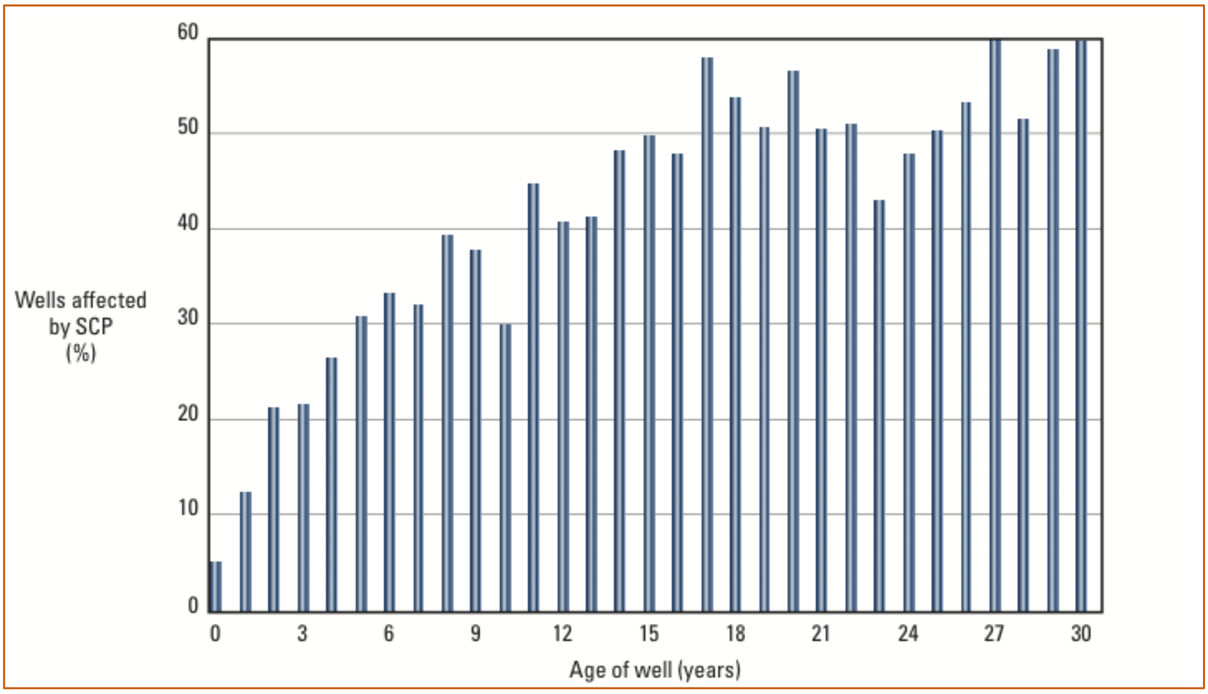

Figure 3.1 – Gulf of Mexico wells with SCP
Figure gathered from Nelson & Guillot, 2006

Failure of the cement sheath to provide zonal isolation can result in annular pressure or flow, SCP or gas migration. Depending on the severity of the leakage, actions must be taken accordingly. It is not uncommon to experience pressure on one of the annuli. In many cases the pressure does not cause a serious problem but is more of an irritation that must be managed.

The pressure must be monitored, and maybe bled off periodically. Reports must be made, risk assessments and possibly permissions to be allowed to keep the well in the current state. Because really there is only one barrier that has been breached.

However, at some stage the barrier needs to be reinstated. Most regulations appear to allow for some leakage (or pressure management) during the wells production life, but require complete isolation for abandonment (AER, 2021; NORSOK, 2021). Then, you might have an annulus that experience a larger more aggressive pressure, in some cases the well must be shut in. This is a more serious issue and if the well must be shut in, an expensive one.

4 Wellbore integrity

Wellbore integrity can be defined as a condition of a well in operation that has full functionality and two qualified well barrier envelopes, by Norsok D-010, any deviation from this state is a minor or major well integrity issue (Torbergsen et al., 2012). A failure of a well barrier element will usually result in reduced well integrity. If a well barrier has failed, the barrier must be restored. In gas wells, wellbore leakage can lead to methane migration into groundwater supplies and/or to the surface where it is an explosive hazard and a powerful greenhouse gas with a warming potential 28-36 times that of CO_2 (EPA, 2020; Stormont et al., 2018).

The long term well integrity is of great importance due to HSE regulations and for potential CO_2 storage along the line. The goal of any CO_2 storage project is to retain 99% of all injected CO_2 in the target area with a maximum surface leakage rate of 0.01-0.1% of CO_2 per year (Carroll et al., 2016). Wells in sequestration projects are generally planned, drilled and completed following best practices, using the best available materials and at the same time following government regulations that were developed to address common problems. As a result, modern wells have a much higher likelihood of well integrity. However, in many areas that are prime candidates for CO_2 storage, there are legacy wells that were drilled when regulations were less restrictive and materials and best practices are now outdated (Carroll et al., 2016).

Wellbore integrity can be compromised by defective wellbore construction, or as a result of chemical and mechanical stresses, such as pressure and temperature, that damage the well during the operation or abandonment phase (Carroll et al., 2016). Potential leakage pathways are illustrated in Figure 4.1 and include interfaces along the casing-cement and cement-rock contacts, fractures and voids withing the cement sheath itself and a failed casing due to corrosion or thread leakage.

There are many possible mechanisms for creating such leakage paths in a wellbore system. Some due to poor completions and some post-completion. According to Carroll et al., 2016 thread leakage between casing joints are possibly accountable for 90% of all tubular failures. Thermal stresses that arise during the operational life of a well or during cement hydration can result in debonding of interfaces and affect the integrity of the cement and thus the well (Carroll et al., 2016; Garcia Fernandez et al., 2019). Flaws introduced in the cement during construction such as mud channels due to poorly displaced mud prior to cementing or gas migration during setting (Carroll et al., 2016; Garcia Fernandez et al., 2019; Stormont et al., 2018; Wigston et al., 2019). Excessive fluid pore-pressures can debond the cement-casing interface (Garcia Fernandez et al., 2019). Additionally, stresses induced on the cement-casing system by the adjacent host rock formation can change, i.e. creep, and create fractures or debond the interfaces (Garcia Fernandez et al., 2019).

However, many mechanisms for creating leakage paths, Bois et al., 2011 analyzed various conditions for microannulus formation, and suggested that the stiffness of the cement and formation may be important considerations in the location and magnitude of microannulus formation (Stormont et al., 2018). They concluded that cement hydration along with wellbore pressure and temperature cycling are of the most important mechanisms for microannuli formation (Stormont et al., 2018).

Casing cement used as a well barrier is an extremely important well barrier element as it has to act as a well barrier both during the operational phase and post operation when the well is plugged and abandoned. In order to ensure integrity, it is crucial for the cement to bond properly to the adjacent formation as well as the casing. To ensure bonding with integrity two tools are used, typically cement bond log (CBL) and a sonic tool (USIT) (Torbergsen et al., 2012). The CBL is also inferring the presence of microannuli, however it does not provide a direct measurement of the microannuli and will generally not be able to detect microannuli less than 100 μm (Garcia Fernandez et al., 2019), meaning that microannuli of size less than 100 μm may not be detected but can still be of issue.

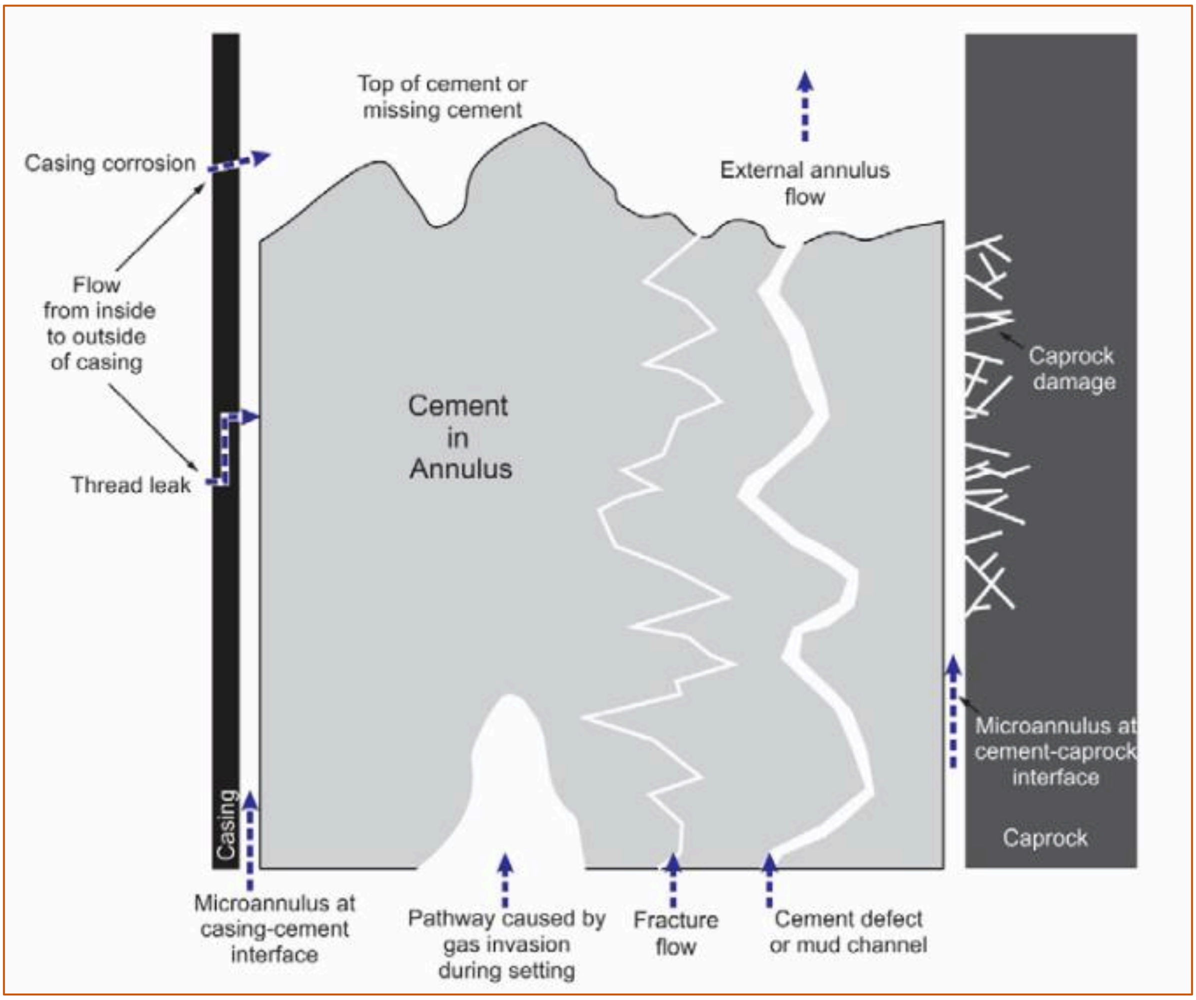

Figure 4.1 Possible leakage pathways in the annulus of a hydrocarbon well
Gathered from Wigston et al., 2019

5 Primary Cementing

Primary cementing is the principal of placing cement slurry in the annular space between the casing and the borehole. After the cement is placed, it is allowed to harden, this forms a hydraulic seal in the wellbore, preventing fluid migration in the annulus. Primary cementing is therefore one of the most critical stages during the drilling and completion of a well. The primary cement job must be planned and executed carefully as there is only one chance to complete the job successfully.

In addition to providing the crucial zonal isolation, the set cement should anchor and support the casing string preventing the adjacent formation to slough or cave into the wellbore. The cement should also protect the steel casing against corrosion by formation fluids. An uncemented steel casing can corrode rapidly when exposed to hot formation brines and hydrogen sulfide (Nelson & Guillot, 2006). It can also be subject to erosion by high velocity produced fluids, especially when solid particles such as formation sand are being transported.

Lateral loads in poorly cemented casing strings can result in buckling or collapse due to the casing being overloaded at certain points. A properly cement casing would on the other hand be subject to nearly uniform loading, approximately equal that of the overburden pressure.

Primary cementing techniques are in principle the same regardless of the casing-string purpose and size. The cement slurry is pumped downhole through the string that's to be cemented, exits the bottom, and displaces drilling mud as it moves up the annulus. Details vary from casing to casing and there are several techniques for acquiring a satisfactory primary cementing. Figure 5.1 illustrates a typical deep well, consisted of a conductor, surface, intermediate and production casing. For a shallower well the number of casing stages is reduced.

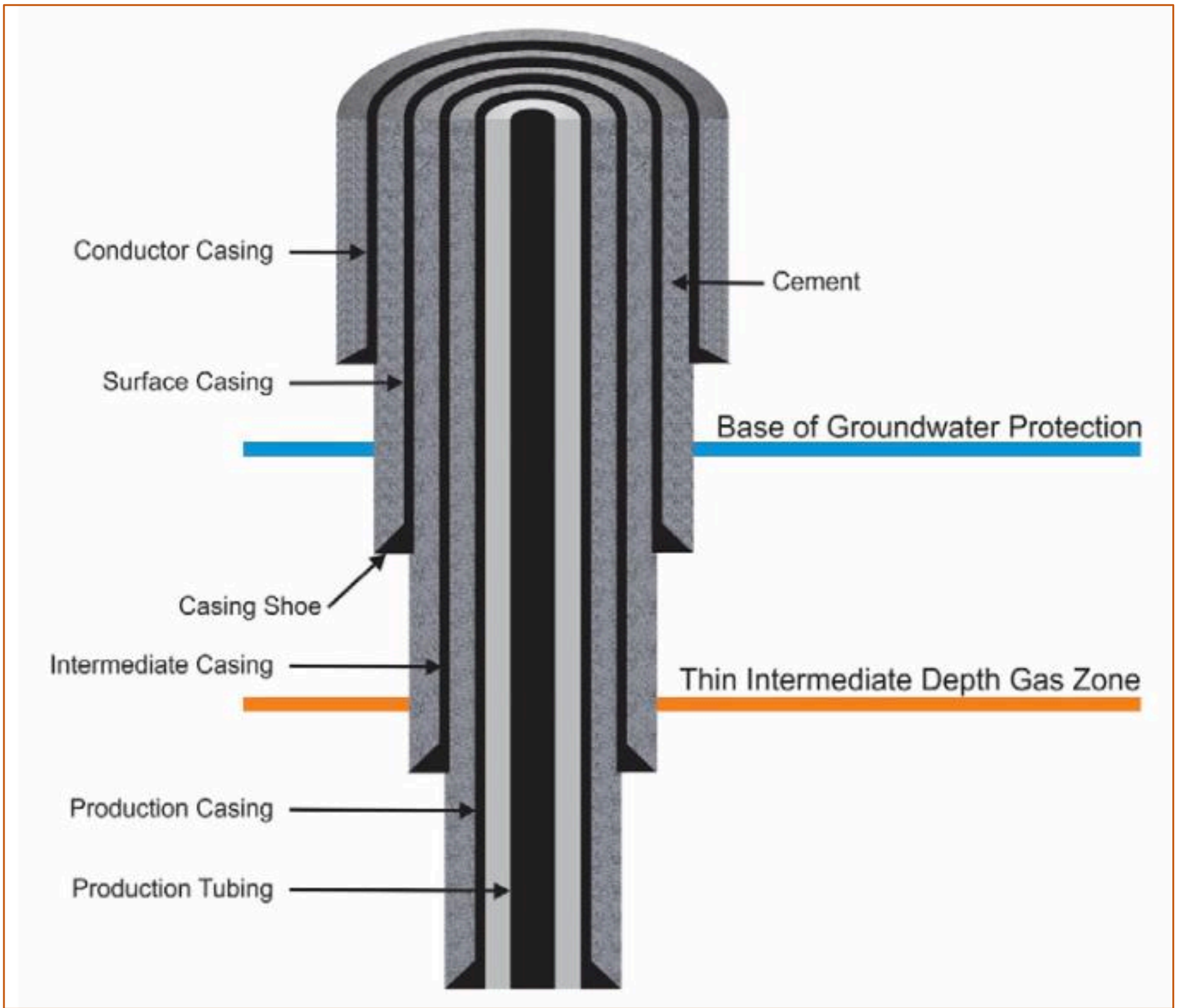

Figure 5.1 Typical well casing diagram
Gathered from Wigston et al., 2019

6 Zonal Isolation

The main goal of the cement is to create a complete and durable zonal isolation. The quality of the cement job has a direct impact on the economic longevity of a producing oil and gas well. From the time the well starts to produce until it is abandoned, the appropriate cement slurry design and placement techniques will affect the productivity physically and economically. Portland cement usually exhibits extremely low matrix permeability if allowed to set undisturbed. Good cement typically have gas and water permeability in the order of 10 μD and 0.01 μD or less respectively, and thus provides a good seal towards downhole fluids (Corina et al., 2021).The cement is however subject to harsh conditions during the productive life, this can affect the longevity of this low permeability.

The first condition is termed "*cracking*", this is caused by thermal or pressure fluctuations in the well caused by the production process. For example, gas wells are subject to large variations in drawdown pressure and temperature as the gas demand changes. Depending on the magnitude and frequency of these production variables, the casing and cement sheath expand and contract in different ways. This causes stress gradients that gradually crack cement, with the subsequent loss of cement integrity and thus an increase in permeability.

Debonding is the second condition. Debonding occurs when the bond between either the cement/rock or the cement/casing interface fails. There are several production practices that can cause debonding:

- The gradual pressure decrease as a well is produced
- Casing movement as subsidence occurs
- Cement shrinkage with time
- Temperature and pressure fluctuations
- Stimulation practices, such as hydraulic fracturing.

Figure 6.1 shows images from a 4-arm caliper log in a well that has experienced severe casing movement. The cement in the region affected by the movement was destroyed, but a good primary cement job kept the unaffected region intact and isolated.

"*Shear failure*" is the third condition, this typically results in complete failure of the cement sheath. Shear failure is normally caused by effective stress increases around a wellbore caused by formation subsidence and movement as the reservoir is depleted. The effect can also occur due to vibrations from downhole pumps or gas-lift operations (Nelson & Guillot, 2006).

Any one of the conditions presented will result in flow paths in the form of discrete conductive fractures in the cement, or microannuli. The paths created and their effective widths create cement permeabilities that far exceeds the intrinsic permeability of the undisturbed cement. Even a small microannuli will result in a larger effective permeability along the cement sheath.

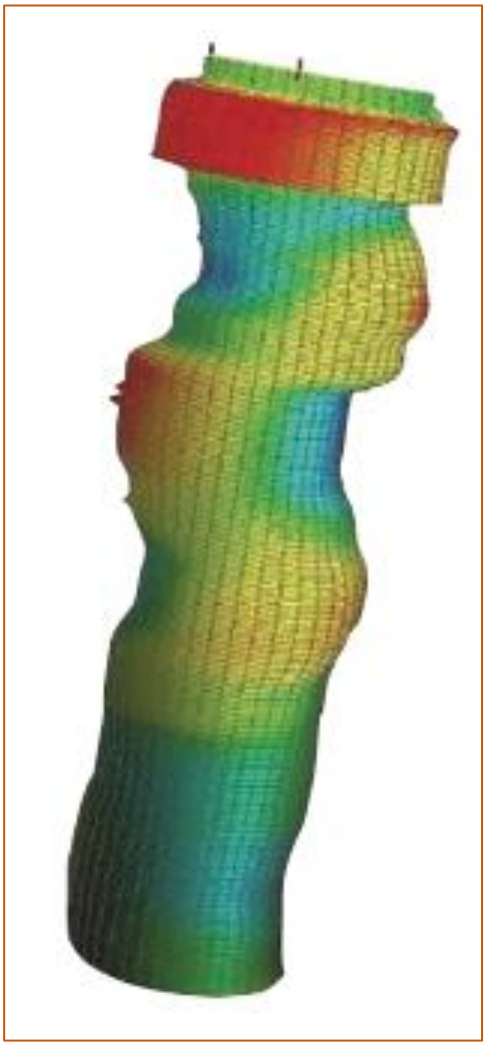

Figure 6.1 – Casing deformation caused by formation movement
Gathered from Nelson & Guillot, 2006

7 Annular Fluid Migration

Annular fluid migration during drilling or well-completion procedures has long been recognized as one of the most troublesome problems of the petroleum industry. It is defined as the invasion of formation fluids into the annulus due to an imbalance in pressure at the formation face (Nelson & Guillot, 2006). The fluids may migrate to a lower pressure zone or possibly to the surface where they pose the greater problem. The most common form of annular fluid migration is gas, and it is by no doubt the most dangerous form. The focus here will be on gas migration, however, most of the concepts presented will indeed apply for other formation fluids.

Gas migration is a problem for both production wells and storage wells. The severity of gas migration varies, ranging from the most hazardous situations, e.g., a blowout due to a serious pressure imbalance during drilling or cementing, to the most marginal, e.g., a residual gas pressure of a few psi at the wellhead. Communication between two or more subsurface zones can occur, in addition to surface related problems. However, such problems are more difficult to detect.

7.1 Practical Consequences of Gas Migration

There are numerous potential consequences of gas migration following primary cementing, but they are not always immediately detectable. Those that manifest themselves at the surface, e.g., SCP or gas flow at the wellhead, may dictate the abandonment of the well. More frequently, remedial cementing is performed until gas flow is stopped and the gas pressure is reduced to a satisfactory level compatible with the operator's policy and local regulations. The efficiency of the remedial cementing is however very poor in instances of gas migration. There are three essential reasons for this: (1) gas channels are difficult to locate, especially if they are less than 1 mm in size; (2) gas channels may be too small to be effectively filled by cement; and (3) the pressure exerted during the remedial cementing operation are often sufficient to fracture the formation or further debond the cement and further worsen the communication problems (Nelson & Guillot, 2006).

7.2 Physical Process of Gas Migration

Gas migration is a complex process and is influenced by several factors. Fluid density, mud removal, cement-slurry properties, cement hydration and interactions between the formation, cement and casing are such factors. Gas communication problems was first recognized in the cementing industry in the early 1960's when gas storage wells in the United States experienced major gas-communication problems (Nelson & Guillot, 2006).

7.2.1 Root Causes for Gas Migration

There are three distinct root causes for annular gas migration, Figure 7.1.

1. The hydrostatic pressure in the annulus falls to a level that is less than or equal to that of the pore pressure of a gas bearing zone.
2. There is sufficient space in the annulus to allow gas entry.
3. There is a path present in the annulus through which the gas can migrate.

All three root causes must be satisfied for annular gas migration to take place (Nelson & Guillot, 2006).

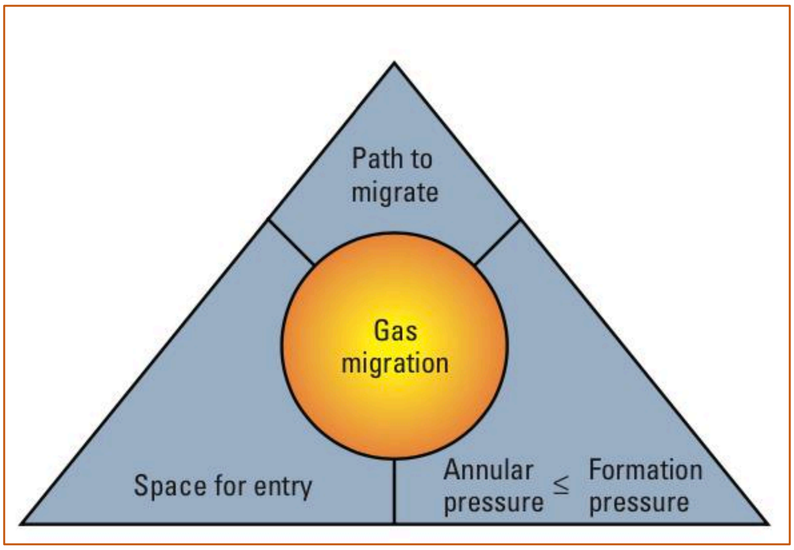

Figure 7.1 Root causes of annular gas migration, all three causes has to be satisfied Gathered from Nelson & Guillot, 2006.

7.2.2 Gas Migration Categories

Gas migration are categorized both by when it occurs and the severity. Figure 7.2 shows how gas migration is categorized by when it occurs.

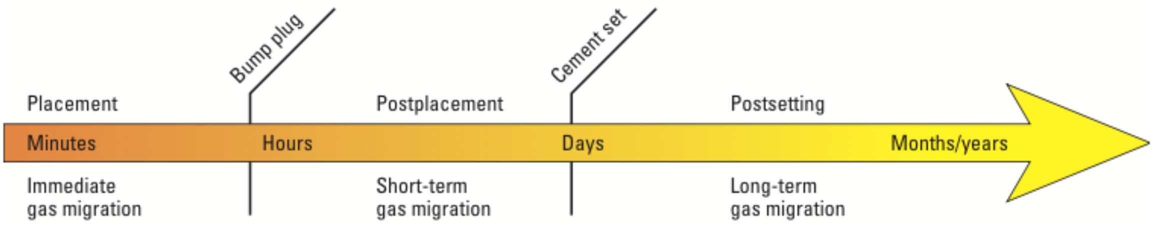

Figure 7.2 – Categories of gas migration by when it occurs Gathered from Nelson & Guillot, 2006

Gas migration are also classified as serious, considered non-serious, or non-serious. There are multiple criteria dictating the severity of the leak, whatever the criteria, serious wells must be remediated immediately to at least a non-serious condition. Non-serious wells must be monitored annually for five years, or until the leakage ceases. Non-serious wells that become serious must be immediately reported and remediated. Non-serious wells that persist past the five-year monitoring period must be remediated at the time of abandonment (Wigston et al., 2019).

7.2.3 Factors Affecting Gas Migration

There are several factors contributing to gas migration, it is important to note that there is no single factor that causes gas migration, but rather a combination of several factors. Factors contributing to gas migration are summarized in Table 7.2.1.

Table 7.2.1 – Factors Responsible for Gas Migration
From Nelson & Guillot, 2006

	Annular Pressure ≤ Pore Pressure	**Space for Entry**	**Path for Migration**
Immediate	Hydrostatic underbalance	Fluid displaced from wellbore	Fluid displaced from wellbore
Short term	Fluid loss	Fluid loss	Slurry permeability
	Gel strength development	Free fluid	Slurry permeability
	Chemical shrinkage of cement	Chemical shrinkage of cement	Filtercake permeability
	Annular bridging	Slurry porosity	Filtercake permeability
	Annular packers	Slurry porosity	Filtercake permeability
Long term	Chemical shrinkage of cement	Chemical shrinkage of cement	Microannulus
		Mud channel	Mud channel
		Free fluid	Free-fluid channel
	Strength development of cement	Dehydrated filtercake	Dehydrated filtercake
		Bulk shrinkage of cement	Bulk shrinkage of cement
			Low cement tops
			Cement sheath mechanical failure

7.2.3.1 Microannulus

The microannulus is referred to the typically very small annular gap or degraded space that may develop between the cement and casing interface (Gasda et al., 2004). The microannulus has been identified as a common leakage pathway in wellbore systems (Gasda et al., 2004; Nelson & Guillot, 2006). The microannulus is a common occurrence and can result from any number of events during the life of a well. However, specific mechanisms for the formation of microannuli include cement shrinkage and/or temperature and pressure changes within the casing (Bois et al., 2011; Carroll et al., 2016; Gray et al., 2009).

Nelson & Guillot, 2006 gives an example of a situation where both temperature and pressure reduction can occur. When the casing is closed in after a cement job, the exotherm generated by the cement hydration will cause thermal expansion of the steel casing. Fluids trapped inside the casing will heat up, causing further thermal expansion. When the casing is eventually opened at a later stage, after the cement has set, the pressure inside of the casing will drop. The heat generated by cement hydration will also eventually dissipate. The casing diameter will consequently return to its original size. Finally, a bulk volume reduction of the cement sheath owing to the chemical shrinkage may result in a microannulus.

Laboratory test of casings set in a cement sheath has demonstrated that microannuli can be formed by pressure cycling (Boukhelifa et al., 2004; Goodwin & Crook, 1992) and thermal cycling within the casing (De Andrade et al., 2015; Vrålstad et al., 2015). It has long been known that a pressure decrease inside the wellbore after the cement is set will result in the diameter of the casing size to reduce. The reduction of the casing diameter may lead to creation of a microannulus. This commonly occurs when the density of the fluid inside of the casing is reduced after the cement job. A wellbore temperature decrease would also reduce the casing diameter.

7.2.3.2 Cement shrinkage

Cement shrinkage contributes to gas migration by causing an annular-pressure reduction and providing space for the gas to enter the wellbore. Cement shrinkage is a result of chemical, autogenous and drying shrinkage phenomena (Kamali, Khalifeh, Saasen, et al., 2021). The hydration process of cement is associated with shrinkage because the volume of the hydration product is less than the volume of reactants (Henkensiefken et al., 2009). The decrease of volume due to the hydration process if referred to as chemical shrinkage. The process of cement hydration continues as the cement slurry hardens. The unreacted cement consumes the remaining water in the pores of the cement, leaving the pores empty. The consummation of the remaining pore water results in a volume reduction, caused by capillary pressure development and extra tension within the cement matrix (Kamali, Khalifeh, Saasen, et al., 2021). The process is simplified in Figure 7.3. The volume change caused by the consumption of pore water is known as autogenous shrinkage. As the cement sets, the volume change is due to autogenous shrinkage, which is lower compared to chemical shrinkage (Henkensiefken et al., 2009). The shrinkage-induced tension can be intensified at the cement sheath inner and/or outer circumference by external loads from pressure or temperature from nearby environments. If the summation of tensions exceeds the tensile strength of the cement, there is an significant increase in risk of forming radial cracks or debonding of the cement sheath (Kamali, Khalifeh, Saasen, et al., 2021). According to American Concrete Institute, ACI, , the internal volume reduction associated with the hydration reactions in a cementitious material is typically 6 to 7 mL/100g of fully hydrated cement.

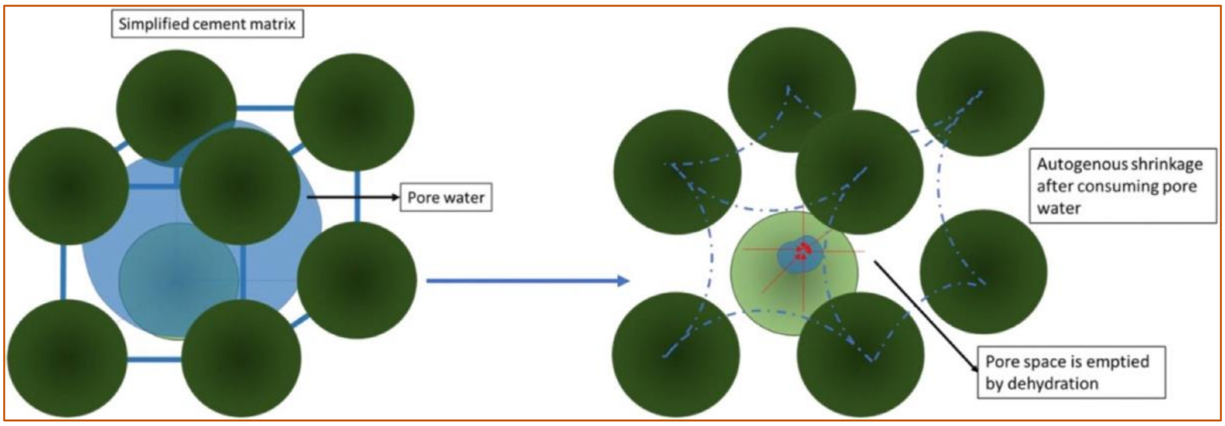

Figure 7.3 – Autogenous shrinkage of the cement-based materials during solidification Gathered from Kamali, Khalifeh, Saasen, et al., 2021

8 Remediation strategies

8.1 Remedial cementing

Remedial cementing is a general term which is used to describe the operations that employ cementitious fluids to cure a variety of wellbore problems. Problems that may require remedial cementing can occur at any time during the life of a well, from well construction to well stimulation, production and abandonment. The term remedial cementing is commonly divided into two broad categories: *plug cementing* and *squeeze cementing*, where the latter is of interest in this study. Squeeze cementing consists of forcing cement slurry through holes, splits or fissures in the casing/wellbore annular space.

During well construction, remedial operations may be required to maintain wellbore integrity during drilling, to cure drilling problems, or to repair defective primary cement jobs.

Remedial cementing is often the only viable option for repairing defective primary cement jobs, to either allow further drilling to proceed or to establish adequate zonal isolation for efficient well production.

8.1.1 Squeeze cementing

Squeeze cementing is the process of forcing a cement slurry under pressure through holes, splits, or fissures in the casing/wellbore annular space. When the cement slurry is forced against a permeable formation, the solid particles in the slurry will filter out onto the formation face as the cement filtrate enters the formation matrix. The slurry is pumped until the wellbore reaches a pre-determined pressure level. An impermeable filtercake, not the setting of the cement, allows the well to withstand this increased pressure. When highly permeable media are present, such as gravel packs, the entire cement slurry may invade the porous medium (Nelson & Guillot, 2006). A properly designed squeeze job causes the resulting filtercake to fill the opening(s) between the formation and the casing. The filtercake forms an nearly impenetrable solid upon curing (Suman and Ellis, 1977 cited in Nelson & Guillot, 2006). In cases where the cement slurry is placed into a fractured interval, the cement solids must bridge the fracture and then develop a filtercake on the fracture face. Most of the slurry is forced into the formation or behind the casing during a squeeze cementing treatment, shown in Figure 8.1.

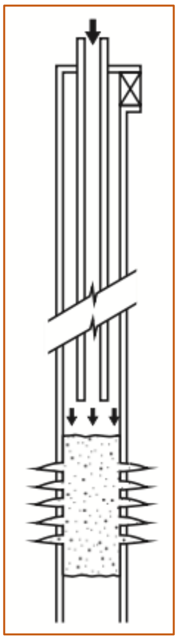

Figure 8.1 – Squeeze cementing, most of the slurry is forced into the formation or behind the casing. Figure gathered from (Nelson & Guillot, 2006)

8.1.2 Reasons for squeeze cementing failures

Whenever a squeeze operation fails to meet the objectives, it is important to analyze the operation as to why it rendered unsuccessful. Squeeze failures may originate from misconceptions of what a squeeze treatment is and what actually happens down hole.

8.1.2.1 Improper slurry design

The cement slurry does not penetrate the pores of the rock. Only the mix-water and dissolved substances penetrate the pores, while the solids accumulate at the formation face and form the filtercake. It would require a permeability of 100 D for solids from a conventional Portland Class G cement to penetrate a sandstone matrix (Nelson & Guillot, 2006). The slurry is only able to penetrate a formation through fissures, fractures, and large holes, these will be referred to as apertures from now. However, the effectiveness of the penetration depends also on the size of the defects in the formation. A common criterion for cement-based slurry, grout fracture penetrations is that the aperture to be filled must be at least three times the size of the largest particle present in the grout (Eklund & Stille, 2008).

8.1.2.2 Excessive final squeeze pressure

A high final squeeze pressure does not increase the chance of success. It does, however, increase the chance of fracturing the formation and losing control of the cement-slurry placement. Once a fracture has been created, an increase in pressure may extend the fracture across various zones, and open unwanted communication between previously isolated zones.

8.2 Current practices and Regulations

Regulations governing the repair of leaky wells are often not prescriptive. They describe broad objectives, and do not specify details on the remedial process. This is reasonable considering that each well is unique and by the thousands. Regulations tend to focus on the classification and reporting requirements rather than potential risks or repair methods. Regulations are often specific to the local jurisdiction and the type of well (e.g., onshore versus offshore). Most regulations appear to allow for some leakage during the well's production life, but require complete isolation for abandonment (AER, 2021; NORSOK, 2021).

The process of repairing annular leakage of hydrocarbon wells generally involve four steps: (1) identifying the source and a suitable depth for intervention; (2) developing communication to the source of leakage/leakage path; (3) sealing the leak; (4) verifying operational success. In most traditional methods, communication with the casing-casing or casing-formation annulus is required in order to inject a sealant to remediate the leak. Communication is commonly established by perforating the casing and typically also penetrating the cement sheath and the near wellbore formation layer. Perforation methods are many, jet perforation with shaped explosive charges, bullet perforation, abrasive jetting, and high-pressure fluid jet perforation are such methods.

Squeeze placement, often referred to as squeeze cementing is the most common method of placing the sealant material. The application of an overbalanced pressure on the sealant forces it into perforations, annuli and voids. With Portland cement, the pressure against a permeable formation results in leak-off causing the cement slurry to dehydrate, building an impermeable filter cake.

8.2.1 Problems with Current Operational Practices

The practice of squeeze placement of sealant materials, such as fine particle Portland cement is generally well developed. However, remediation of the annular cement sheath have often proved difficult. Saponja, 1999; Wojtanowicz et al., 2001 noted that squeeze cementing success rates were as low as 50% for Husky's wells in the Lloydminster region of Alberta and for offshore wells in the Gulf of Mexico, respectively. Anecdotal evidence suggest that the cost of remedial cementing can vary from as low as tens of thousands of dollars to millions or tens of millions of dollars on problematic wells (Wigston et al., 2019). Inadequate identification of the source and poor communication with the annular leak path are two of the main reasons for unsuccessful remedial operations. Remediation materials and methods are closely linked to well abandonment, as in order to abandon a well surface casing vent flow (SCVF) or SCP must be identified and eliminated.

9 Experimental methodology

As remedial treatment using OPC often proves unsuccessful as it is unable to penetrate the leakage path effectively due to the particle size, low-viscosity thermosetting polymers (resin) are a viable option. Thermosetting resins are particle-free fluids which solidify into an impermeable material upon curing. The curing process is temperature-activated and occurs at a predefined temperature. The viscosity and density of the resin can be tailored for various applications by addition of particles (Vrålstad et al., 2019). Resins have been used in the North Sea as well as Gulf of Mexico as a plugging material (Beharie et al., 2015; Davis, 2017 cited in Vrålstad et al., 2019). Thermosetting resin have also been used as a squeezing material to regain zonal isolation between two casings (Al-Ansari et al., 2015). Five samples comprised of steel casings filled with API Neat class G cement are subject to leakage testing to establish the permeability. The samples are subsequently subject to remedial treatment using a thermosetting polymer, ThermaSet®.

9.1 Samples

9.1.1 Preexisting samples

Samples 2 and 3 were provided by Mohammadreza Kamali for testing, they have previously been used in Kamali, Khalifeh, Eid, et al., 2021. The samples are comprised of API Neat class G cement, 44% by weight of cement (BWOC) de-ionized water, cured in 4140 steel casings. The steel casings are originally established with three holes at the center of the body with an orientation of 120 deg. These holes are plugged, as seen in Figure 9.3, for the flow measurements conducted in this experiment.

9.1.2 Construction of New Samples

Three more samples were constructed during the project to have a broader reference point for testing, samples 4, 5 and 6. The specimens were constructed out of hollow carbon steel casings, St52-3, with dimensions OD 51.50 mm, ID 37.30 mm and a total length of 120 mm (axial). The carbon steel casings were supplied by NORCE. The casings were cleaned thoroughly before being filled with cement. The cement used was 700g dry weight of API Neat class G mixed with 44% BWOC de-ionized water according to API 10B. The slurry was mixed using the API high-speed mixer Waring blender, where the mixer starts to mix the slurry for 15 s at 4000 rpm and continues mixing for 35 more seconds at 12,000 rpm.

The cement slurry was heated to bottom-hole circulating temperature (BHCT) which was selected to be 65 ˚C. The temperature ramp-up rate was 1 ˚C/min. After reaching BHCT, the slurry was pre-conditioned for 30min. Atmospheric consistometer, OFITE Model 60, was used for the pre-conditioning of the cement slurry.

The steel casings were filled with approximately the same amount of cement, topped off with water, and taped off top and bottom as to not lose the cement. The samples were then placed in a pressure vessel and cured at 90 ˚Celsius at 2200 psi over a 7-day period. The samples were then allowed to cool down to ambient temperature to avoid inducing thermal shock to the system. The samples were created in a similar manner to best mimic Samples 2 and 3. Neither of the samples were constructed with any artificial flaw or microannuli.

The samples were stored in Ziplock bags until testing as to keep as much humidity as possible in the sample.

9.2 Flow measurements

The specimens were subject to atmospheric confining pressure and sealed at the top and bottom using steel endcaps, fabricated by NORCE, with an integrated port top and bottom for flow measurements. Water flow measurements were made along the axis of the specimens and thus would include any potential microannuli and went into a wastewater container. The test system is shown in Figure 9.1. The piping was made as short as possible to reduce the potential pressure drop over the length of the pipe. A pressure gauge mounted just before the sample showed negligible pressure drop.

Distilled water was pumped using Quizix QX series pump with constant pressure and thus regulating the flowrate accordingly. Water was introduced into the sample using the topside port and flowed through the sample, including the cement-casing interface. The sample was oriented on a flat surface and thus did not include any elevation change. The flowrate and pressure were read from the pumps interface.

9.2.1 Permeability measurements

Permeability was interpreted from water flow measurements using Darcy's Law

$$k = \frac{Q\mu}{\nabla P A} \tag{1}$$

where k is the permeability, Q the volumetric flowrate, μ the fluid viscosity, A the cross-sectional area involved in the flow and ∇P the pressure gradient.

Darcy's law states that the flux changes linearly with the change in pressure difference. With the test conditions used linear flow occurred, the flux varied linearly with the pressure gradient and thus Darcy's law is valid. The flow measurements were done in several pressure steps while logging the respective flowrate. Once the flowrate was considered stable the measurements were averaged over the last 20 minutes of the run and noted as the respective flowrate. The flowrate at the different pressure stages were then plotted and one could find the slope and thus the permeability of the sample.

Once the permeability values were obtained, the cubic law (Equation 2) was used to interpret the permeability as a hydraulic aperture of the microannulus.

$$h_a^3 = \frac{12kA}{w} \tag{2}$$

Where h_a is the hydraulic aperture, k is the permeability of the wellbore system estimated from the observed leakage, A is the cross-sectional area of the entire specimen and w is the length of the hydraulic aperture which, if flow is assumed to occur thorough cement-casing microannulus, can be approximated by the circumference of the inside diameter of the steel casing.

Use of this equation implies that all of the flow passes through the microannuli. This assumption is based on the work done by Garcia Fernandez et al., 2019; microannuli have a transmissivity many orders of magnitude higher than that of intact cement. It is also assumed that the flow between two parallel plates is equivalent to the flow between two concentric cylinders; this assumption produces negligible error (Stormont et al., 2018).

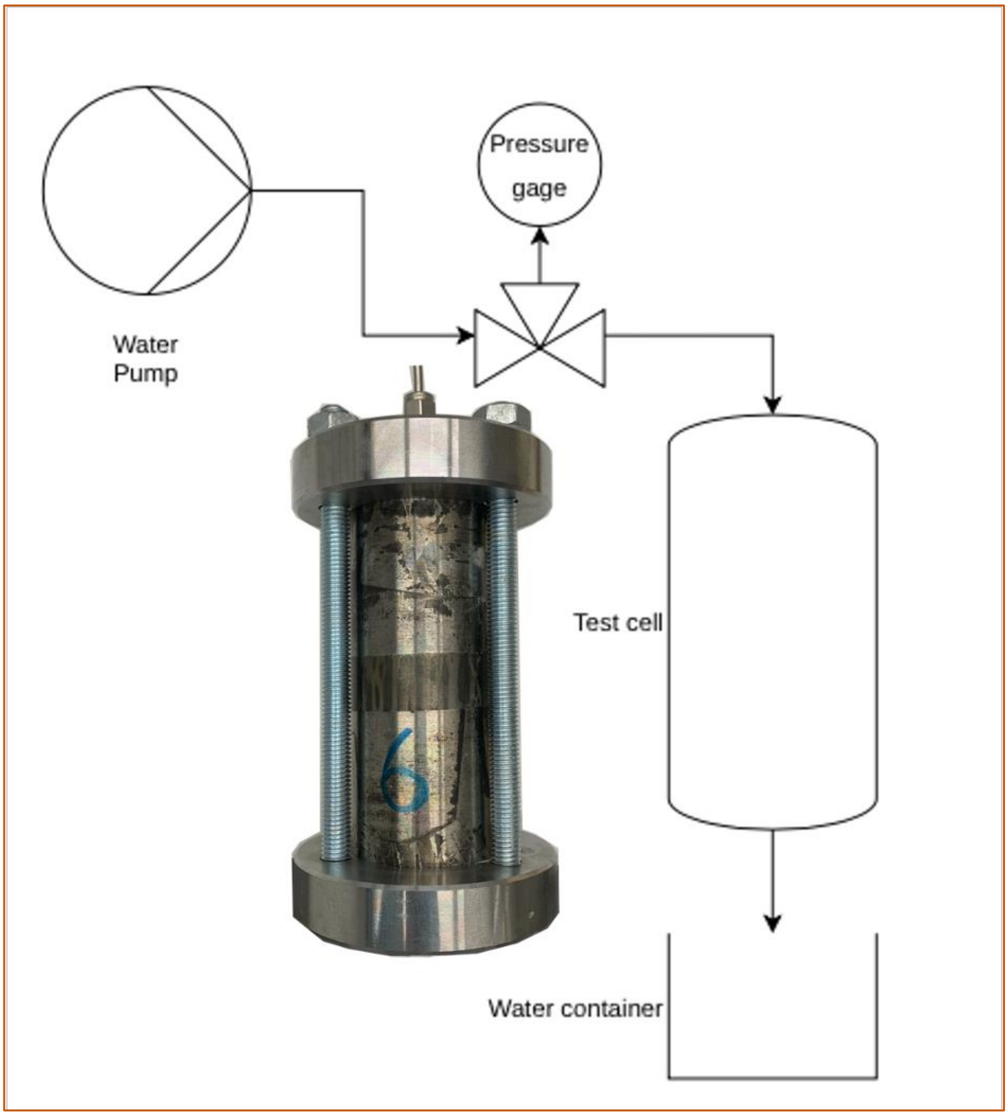

Figure 9.1 Test system used for flow measurements

9.3 Treatment

At the conclusion of the flow measurements, the samples were tried injected with thermosetting resin, ThermaSet®, provided by Wellcem AS. The resin based of vinyl toluene was mixed according to the manufacturer's recipe at 600 rpm using Heidolph overhead stirrer model Hei-TORQUE. The setup used during the treatment process was similar to that of the flow measurements (Figure 9.1), where the setup differs is that the pump was connected to a piston cell filled with the resin which was then connected to the sample. The water displaced the piston in the cell pushing the resin into the connected sample. The samples are water-wet in this process.

If the sample produced a measurable volume of resin during injection it would prove successful. However, if the sample did not produce a measurable volume of resin, the injection would prove unsuccessful.

The samples were subject to two different recipes of ThermaSet. One of which was optimized for plugging and one for squeezing. The reason for this was simply convenience, trial and error. The ThermaSet optimized for plugging was already at the University of Stavanger. The resin had previously been used in Kamali, Khalifeh, Saasen, et al., 2021. However, the resin had not been tried to inject and thus it was uncertain if the resin would prove suitable to this experiment. The ThermaSet which was optimized for squeeze treatment was provided to be compatible to inject into the very tight samples that were subject in this experiment.

Viscosity was measured for both version of ThermaSet. The ThermaSet optimized for plugging experienced a viscosity of around 80 cP and the ThermaSet optimized for squeezing 40 cP. The viscosity measurements were made using OFITE Model 900 Viscometer following API 10B-2 Recommended Practice (API, 2013).

N-heptane Sudan Blue II dye was mixed into the ThermaSet upon injection. The ThermaSet cures into yellow/white as seen in Figure 9.2, the dye was mixed into the resin in an attempt to make the color more vibrant for a later study. It is important to note that the dye was not recommended by the manufacturer of the resin nor was it said to be compatible with the resin by the manufacturer. The dye was readily available and was thus used in the study. The dye was believed to be relatively new, however, after further investigation the dye is between 30 and 40 years old. It is uncertain if the chemical composition of the dye is altered after all these years and could cause problems.

Figure 9.2 – Sample 4 after finished testing, the resin has cured into yellow/white. For illustration purposes.

Figure 9.3 – Ports on the side of samples 2 and 3, here fitted with blinds for testing

10 Results and discussion

10.1 Flow measurements

The flow measurements are summarized in Table 10.1.1; Figure 10.3; Figure 10.4. It is clear from Table 10.1.1 that all samples experience leakage as the permeability are several orders higher than what is considered for intact cement. However, the degree of leakage varies considerably for all samples. What is interesting to note is that Samples 2 and 3 are of the same origin, and Samples 4, 5 and 6 are of the same origin. For Sample 4, 5 and 6 the permeability ranges from 3.498 μD to 708.663 μD. From section 6 Zonal Isolation, the typical permeability value of an intact cement matrix is as little as 0.01 μD for water. This indicates that the samples experience some form of flaw within the cement sheath itself or the cement-steel interface, a microannuli. Stormont et al., 2018 concluded that one of the most important mechanisms for microannuli creation was cement hydration. Given the experimental methodology of the samples, cement hydration appears to be the source of leakage through microannuli and/or any flaw in the sheath itself.

During the process of permeability testing, the distilled water (containing CO_2 from the atmosphere) leaving the sample through the outlet experienced an increase in pH, indicating a reaction with the cement, Figure 10.5. The wastewater contained visible $CaCO_3$ further indicating a reaction with the cement (Figure 10.6; Figure 10.7). However, the process of efflorescence is normally not damaging to the cement (Dow & Glasser, 2003).

10.1.1 Sample 2 and 3

Samples 2 and 3 were subjected to a permeability test using the ports on the side prior to this experiment. The flow measurements from that test were not finished and are mostly not of interest. However, sample 2 sheared at 500psi (34.47 bar), dislodging parts of the cement core, Figure 10.9. Approximately 1 cm of the core protruded outside the casing, the protrusion was cut flush with the casing as to fit with the new setup. Upon re-plugging the side port due to leakage through the port, the cement core appears to have collapsed as a result of the force exerted on the cement from the plug and the void, Figure 10.8.

From Figure 10.12 it is clear that the flowrate of Sample 3 follows linear flow up until 45 bar where the sample experience a sudden increase in flowrate at 55 bar. Figure 10.11 shows the flow measurements for Sample 3 without the measurement at 55 bar, and it is clearly linear. The exact reason for the increase in flowrate is uncertain, however, a possibility is the expansion of the casing at the highest pressure, increasing any flaw and/or the microannuli and allowing more flow. The permeability and hydraulic aperture for Sample 3 is based on the measurements up until 45 bar, as the flowrate at 55 bar clearly deviates from the linear trend.

10.1.2 Sample 4, 5 and 6

Samples 4, 5 and 6, however, being from the same batch does experience significantly different permeabilities. Sample 4 has a permeability of 3.498 μD while Sample 5 has a permeability of 284.422 μD. Sample 6 experienced the largest permeability of all five samples, 708.663 μD. However, Sample 6 is believed to have been dropped, which could increase the permeability of the sample due to possible fractures induced on impact. Sample 6's permeability is also based off two measurements as the flowrate exceeded the pumps capacity of 7.5 mL/min at the next pressure stage.

10.1.3 Variation in permeability

The samples experienced large variations in permeability, and the exact reason for this is uncertain. Stormont et al., 2018 conducted a similar experiment using gas, however, the samples created without artificial microannuli in Stormont et al., 2018 were measured to have permeabilities less than 1 μD. The setup used were able to give pore pressures up to 150 bar, whereas the pressures used in this experiment were no larger than 55 bar. The samples created by Stormont et al., 2018 used a different steel casing, different water/cement ration of 0.33 and the samples were stored in a 100% humidity curing room until tested. This indicates that it is possible to create samples without artificial microannuli which experience the proper permeability considering that there is no leakage.

The Samples studied in this experiment were of 0.44 BWOC meaning that the cement is more pliable, however, not as strong as the cement used by Stormont et al., 2018. Another consideration for the variety of permeability is the choice of steel casings. The compatibility of the casing material and the cementitious slurry is a necessity. In an earlier experimental study by Khalifeh et al. [14] cited in Kamali, Khalifeh, Eid, et al., 2021 incompatibility of aluminum pipe and API Neat class G cement was highlighted. Although the casings used in this experiment are all steel casings, there are variations in composition which could lead to different bonding qualities. However, the casing composition is unlikely to have an effect as the spread in permeability for sample 4, 5 and 6 are independent of the casing composition.

The samples constructed in Stormont et al., 2018 measured 203 mm (axial) cured while the samples studied in this thesis measured at most 118 mm (axial). This must also be taken into consideration as any flaw would dominate a larger percentage of the sample. However, samples 4, 5 and 6 are only differed by at maximum 3 mm. This should not give Sample 5 a permeability 80 times higher than Sample 4, and Sample 6 a permeability 200 times that of Sample 4.

The samples were stored in Ziplock bags. It is possible that some of the samples were exposed to more air than others and thus experienced a stronger drying shrinkage. The age of the samples does also affect the amount of drying shrinkage the sample experience, Figure 10.1. Sample 4 and 5 were tested the same day as the curing were finished, while Sample 6 was allowed 12 more days to continue the process of drying shrinkage. This could be a part of the reason for the extremely high permeability of Sample 6.

The samples in Stormont et al., 2018 were stored in a 100% humidity curing room until tested. The samples used in this experiment were stored in Ziplock bags at the laboratory. The humidity in the laboratory was measured to be 67%, however, this could change, and the accuracy of the measurement is uncertain. However, higher humidity results in less shrinkage, (Figure 10.2). This could also explain the extremely high permeability of Sample 6 as it was stored in lower humidity for longer than Sample 4 and 5.

*Table 10.1.1 – Permeability values and hydraulic aperture **before** treatment*

Sample	Fracture Permeability [μD]	Hydraulic Aperture [μm]
2	45.743	2.513
3	0.528	0.568
4	3.498	1.067
5	284.422	4.621
6	708.663	6.265

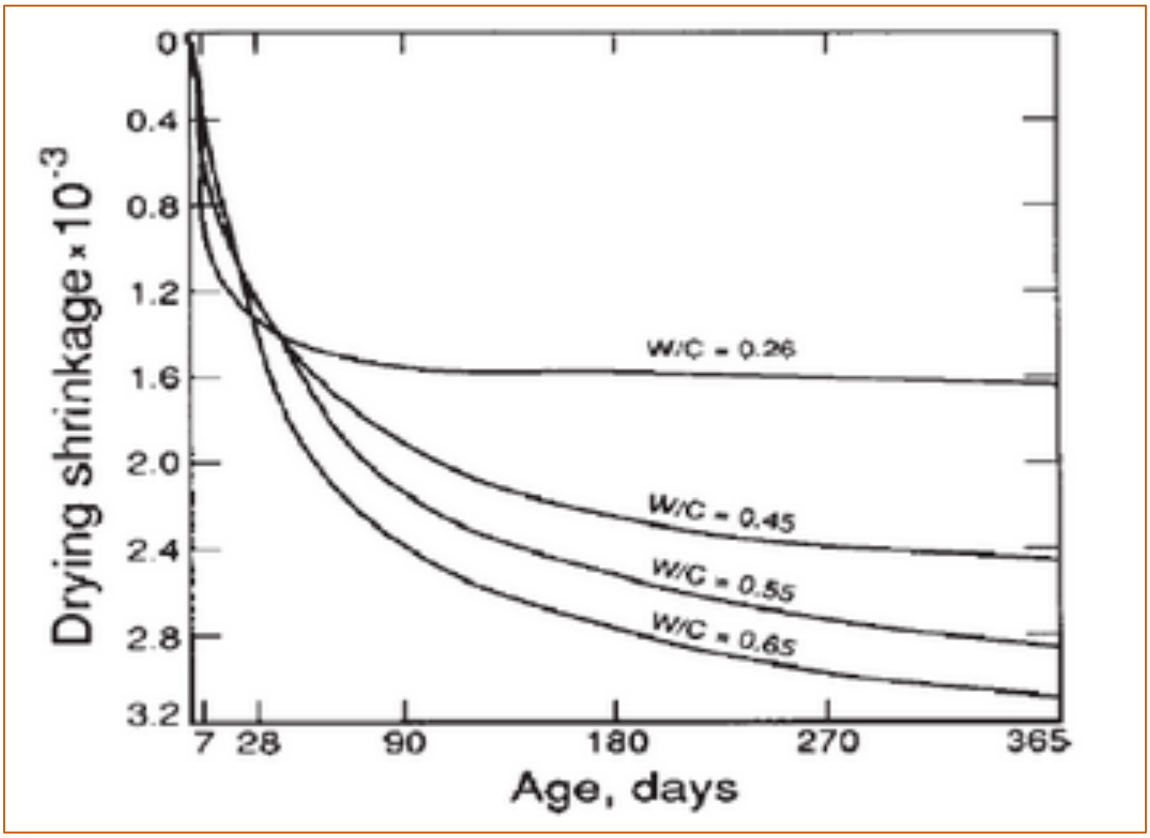

Figure 10.1 – Effect of w/c ratio on the drying shrinkage of concrete as a function of time. Sample 6 would experience a stronger shrinkage due to it being "older" when tested. Data by Hallar (from Soroka, 1993 cited in Idiart, 2009)

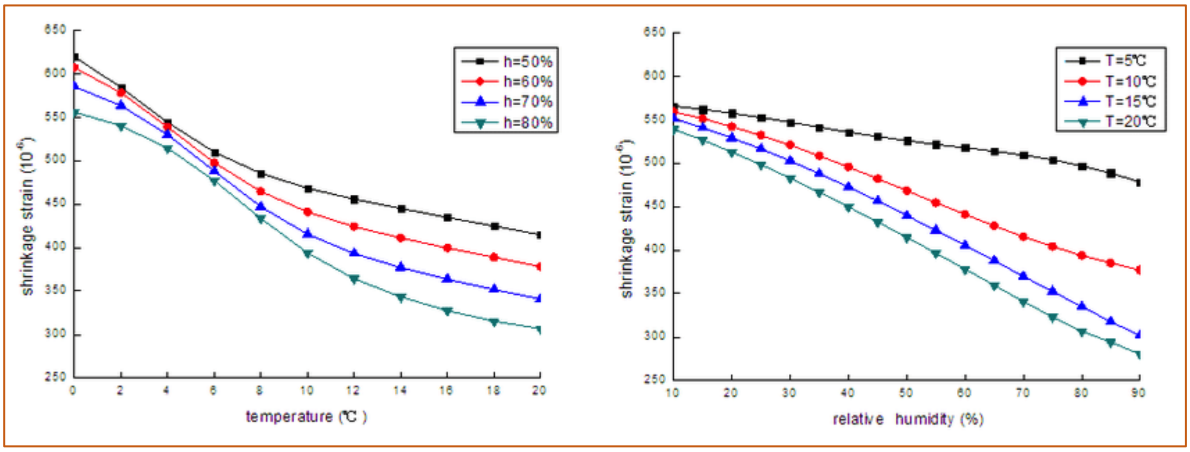

Figure 10.2 – The effect of humidity on shrinkage under different humidity and temperatures. Gathered from Wang et al., 2010

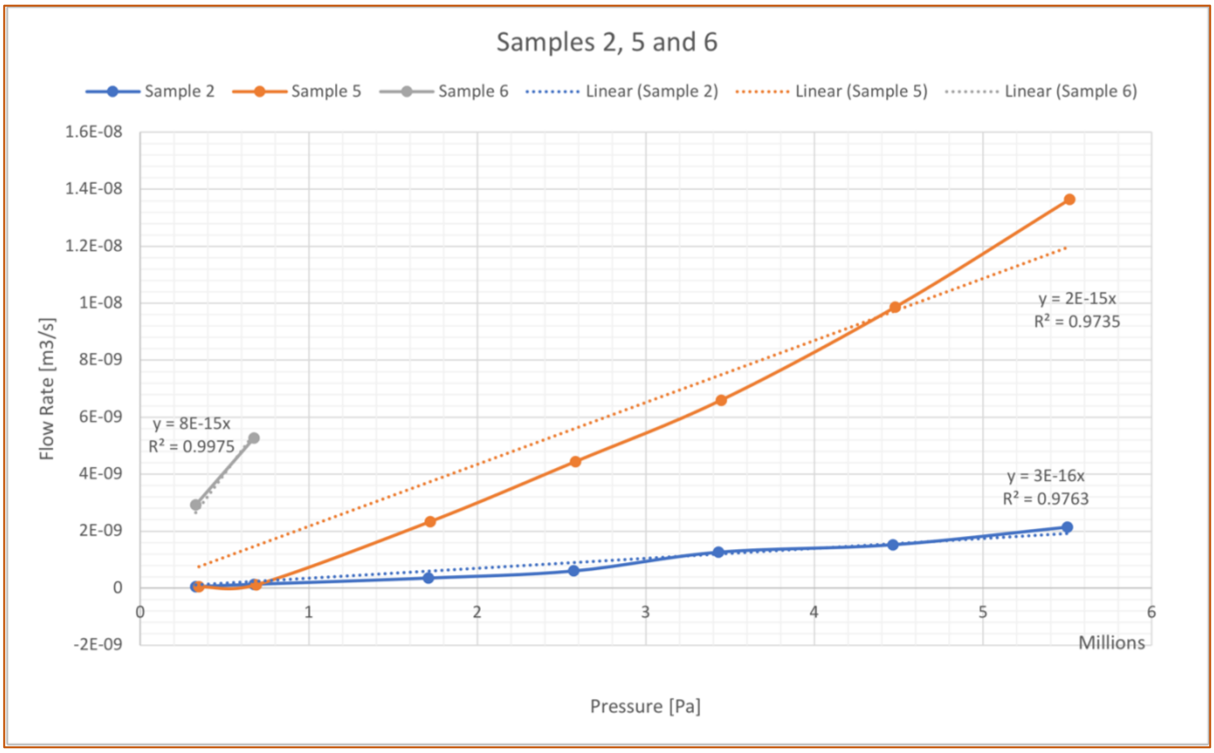

Figure 10.3 – Flow measurements of the most permeable samples 2, 5 and 6.

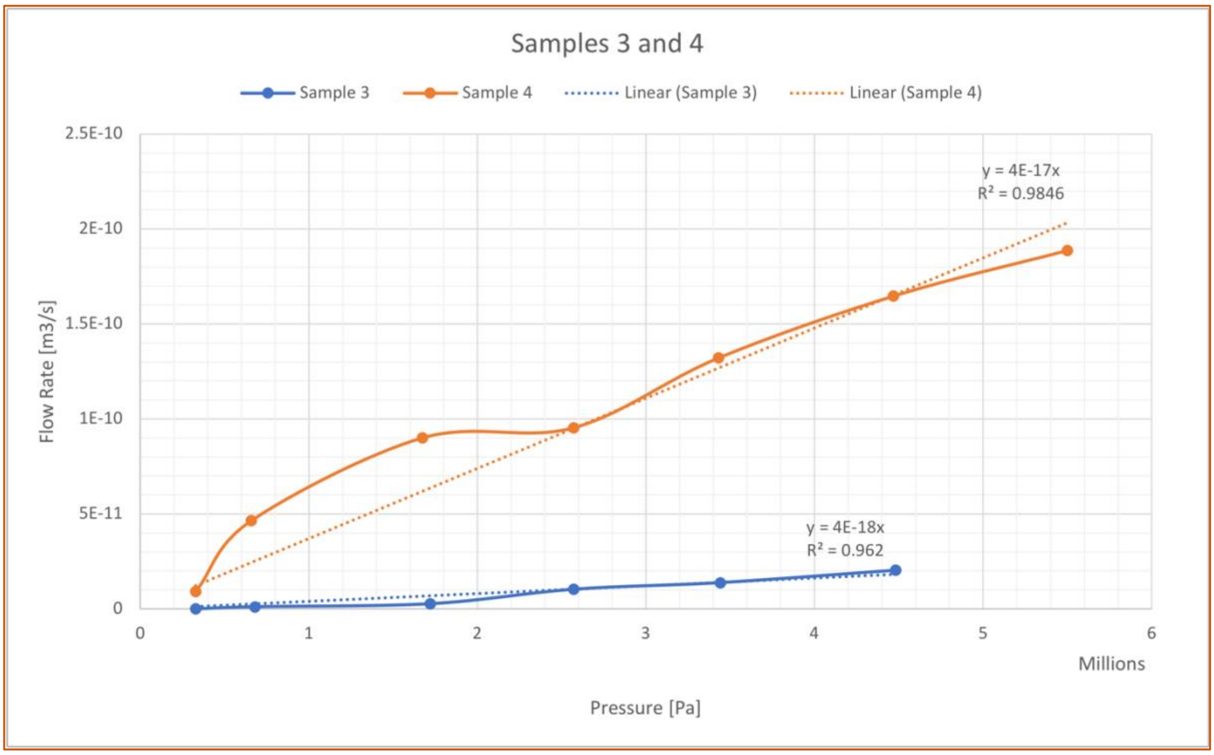

Figure 10.4 – Flow measurements of the least permeable samples 3 and 4.

Figure 10.5 – Distilled water experienced an increase in pH after flowing through the samples

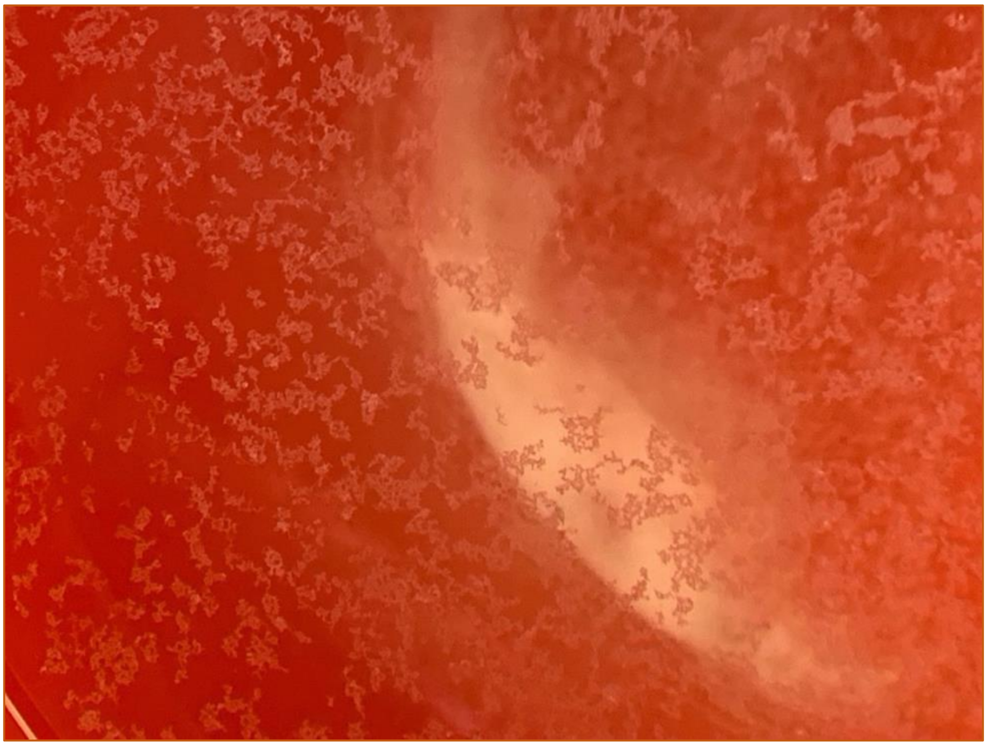

Figure 10.6 – Water from the outlet of the sample, $CaCO_3$ is visible after the water has interacted with the cement

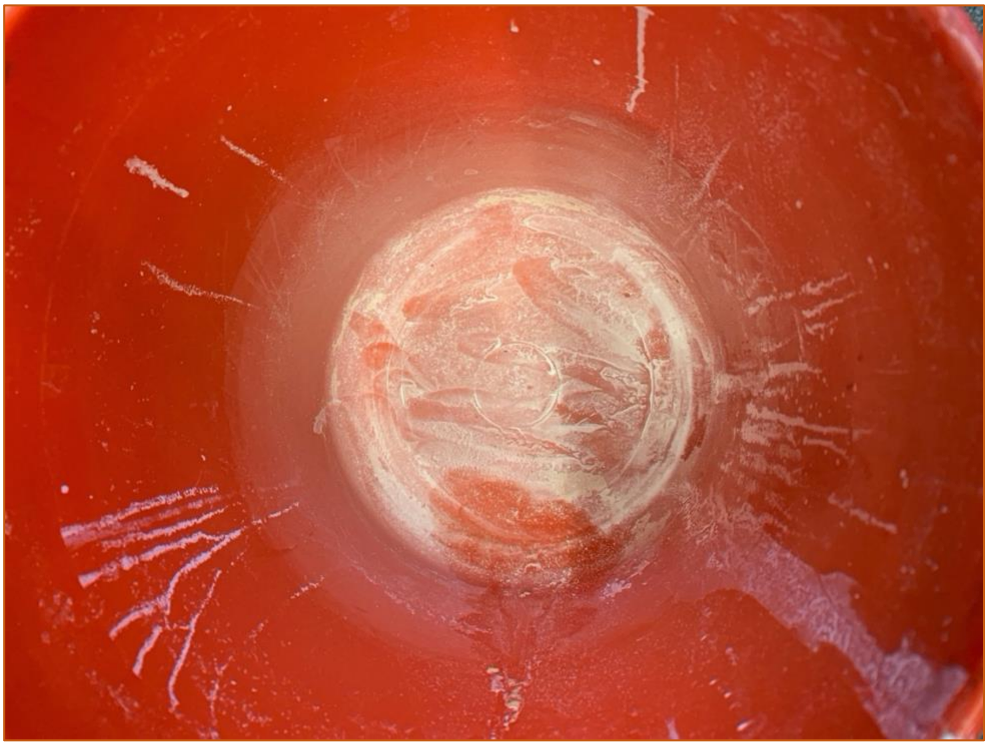

Figure 10.7 - $CaCO_3$ precipitated from wastewater

Figure 10.8 - Sample 2, the cement core appears to have collapsed from the pressure exerted by the blind plugs which was used during flow measurements.

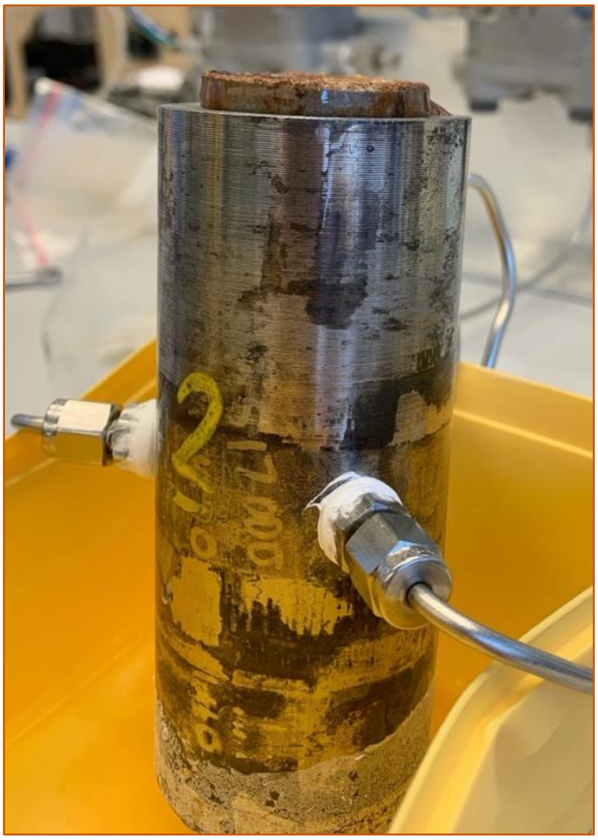

Figure 10.9 – Sample 2. Water injection through the sides. The cement core sheared at 34.47 bar. The sample is in the process of dislodging the core.

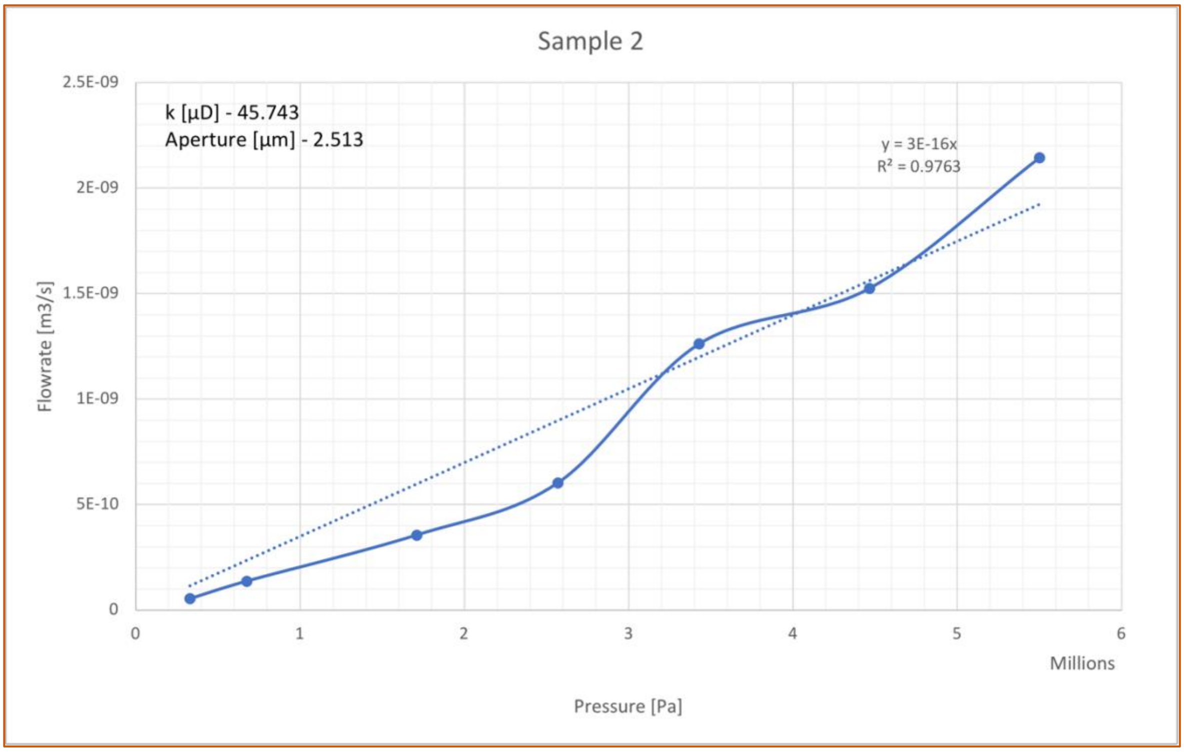

Figure 10.10 – Flow measurements of sample 2

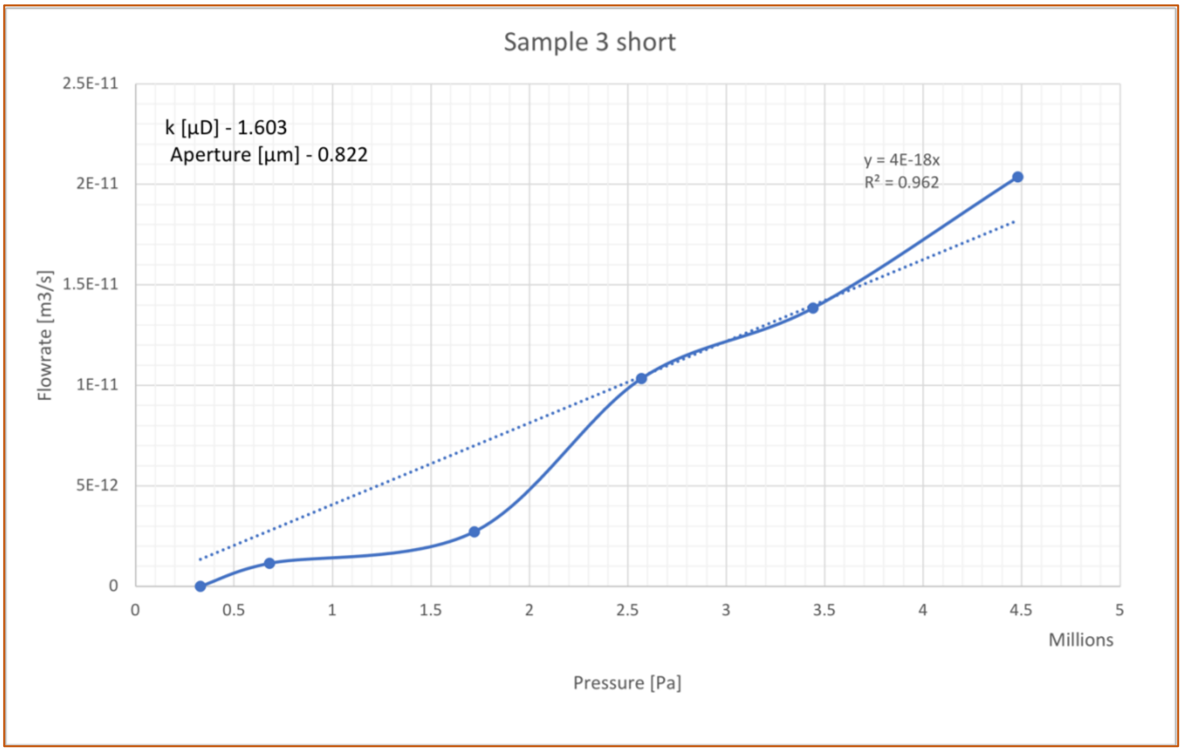

Figure 10.11 – Flow measurements of sample 3 – without 55 bar measurement, clearly this is linear flow.

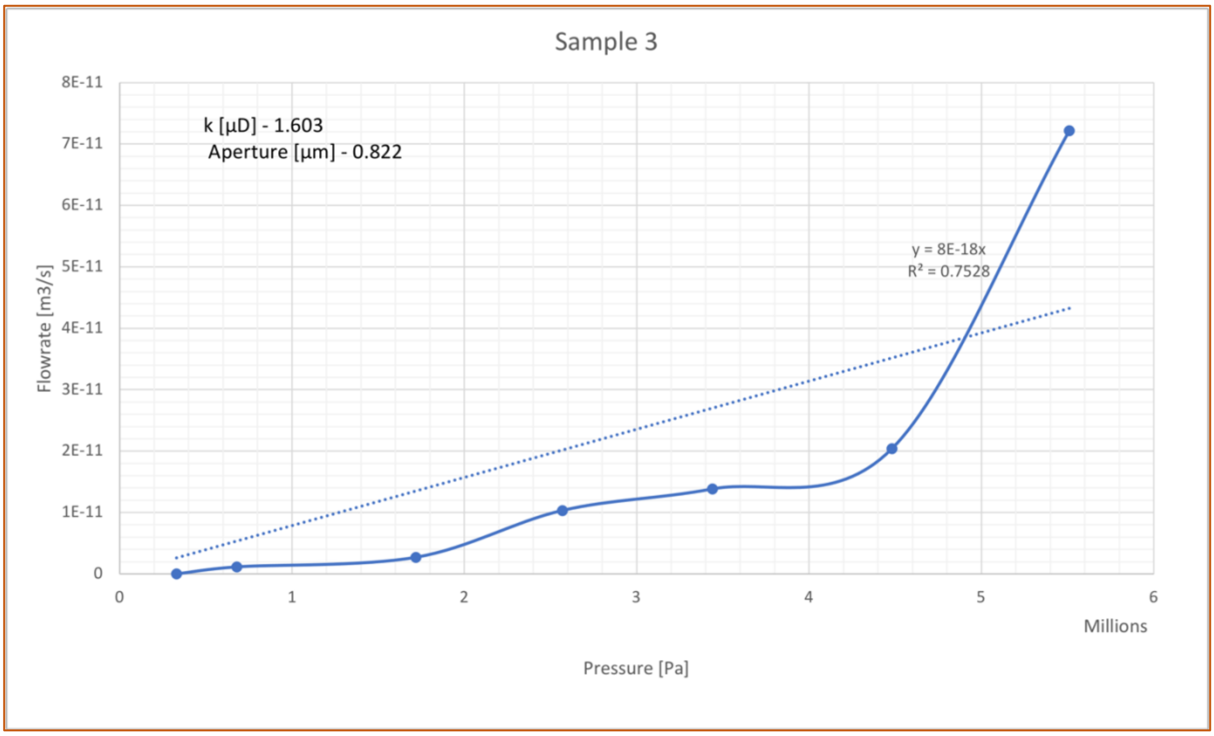

Figure 10.12 – Flow measurements of sample 3, deviation from linear flow

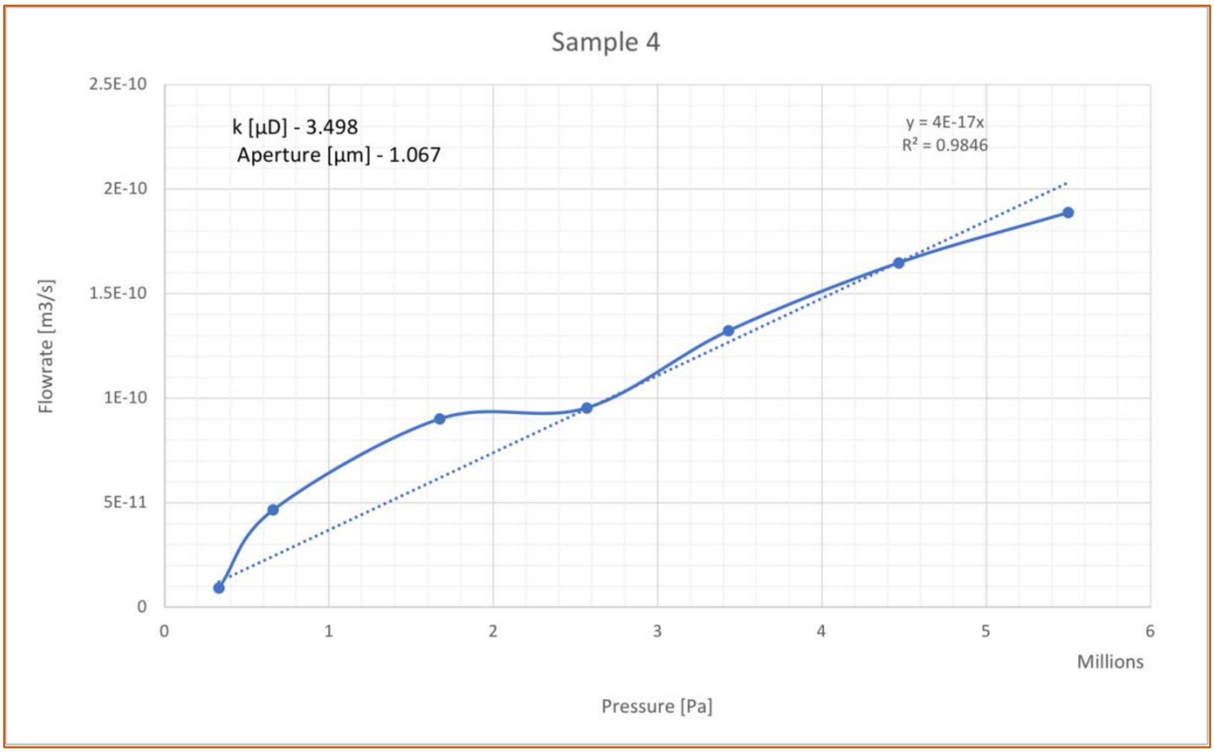

Figure 10.13 – Flow measurements of sample 4

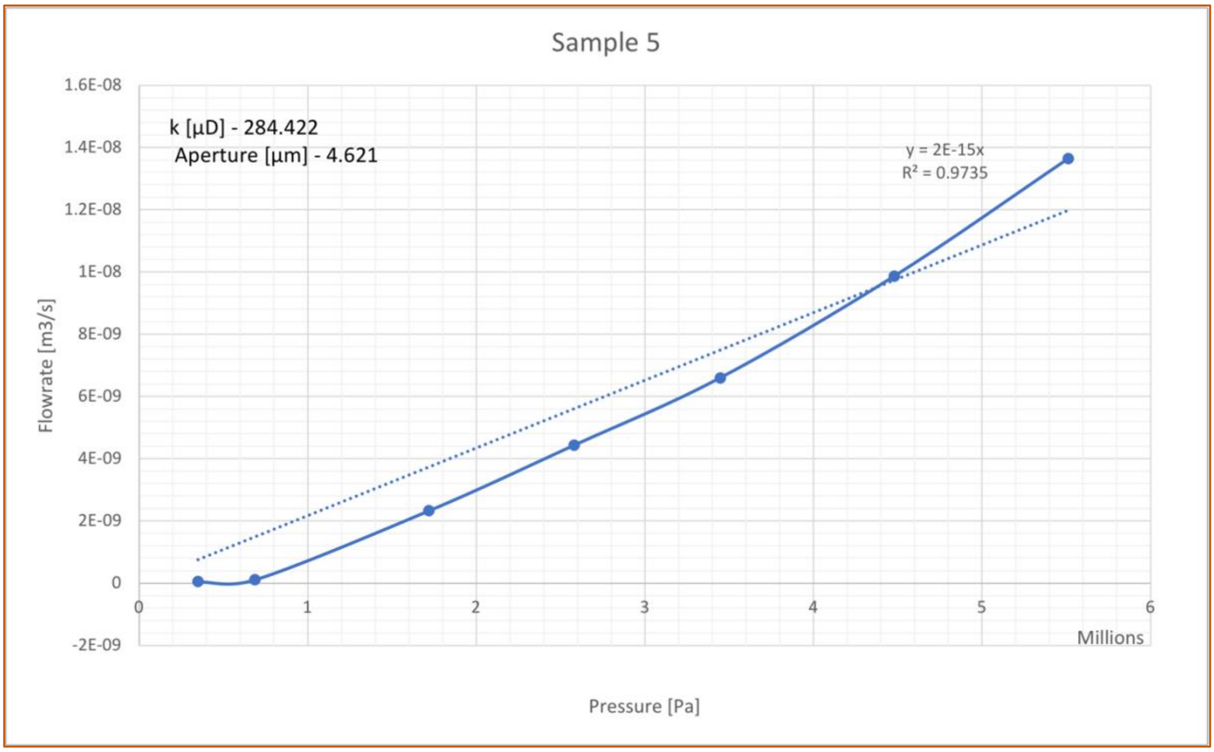

Figure 10.14 – Flow measurements of sample 5

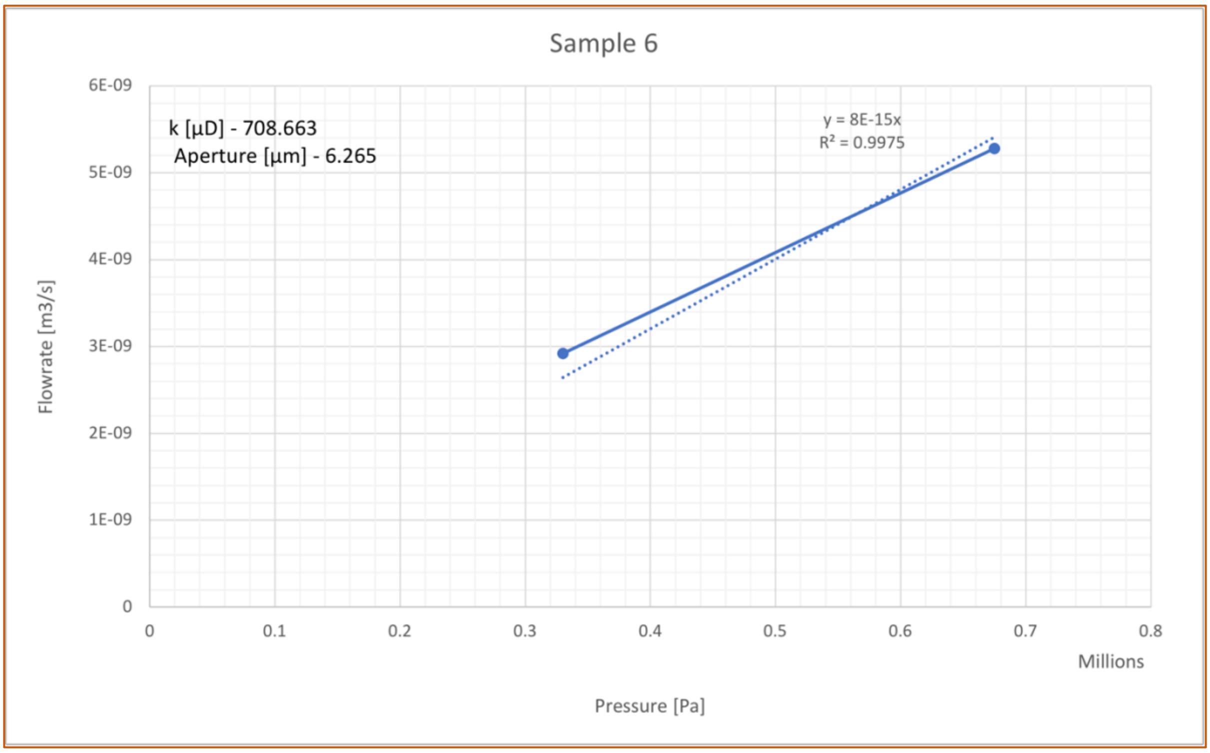

Figure 10.15 – Flow measurements of sample 6

10.2 Treatment

As the samples were treated with different recipes of ThermaSet and some did not contain dye, Table 10.2.1 summarizes the differences for simplicity.

Table 10.2.1 – Treatment material for each sample

Sample	ThermaSet Type	Dye	Produced Treatment Material
2	Squeeze	Yes	Unsuccessful
3	Squeeze	Yes	Unsuccessful
4	Squeeze	No	**Successful**
5	Plug – Squeeze	Yes – No	Unsuccessful*
6	Plug	Yes	**Successful**

* Sample 5 did produce resin and are thus unsuccessful. However, upon inspection Sample 5 experienced altered resin at the bottom of the sample. The resin experienced an increase in viscosity.

10.2.1 Plug ThermaSet

At the time of the first treatment, the ThermaSet optimized for plugging was in house. It was uncertain if it would be able to squeeze into the small apertures of the samples reviewed in this study. The resin was mixed with n-heptane Sudan Blue II in order to cure as a blue/green resin instead of yellow/white. This was done due to possible uses of the samples in a later study.

The resin mixed with n-heptane Sudan Blue II was injected into Sample 6, which proved successful. Sample 6 produced resin. However, for Sample 5 having the second largest permeability, the resin did inject for some time, until the flowrate eventually went to zero. Upon inspection of Sample 5, the resin did flow through the sample. The resin had increased in viscosity and was more gel-like, Figure 10.16. The reason for the gel-like viscosity is unknown and was not answered by the manufacturer. The injection of Sample 6 had proved successful and Sample 5 unsuccessful, only Sample 6 was cured at the time as Sample 5 was uncertain and would be tried reinjected with the resin optimized for squeezing.

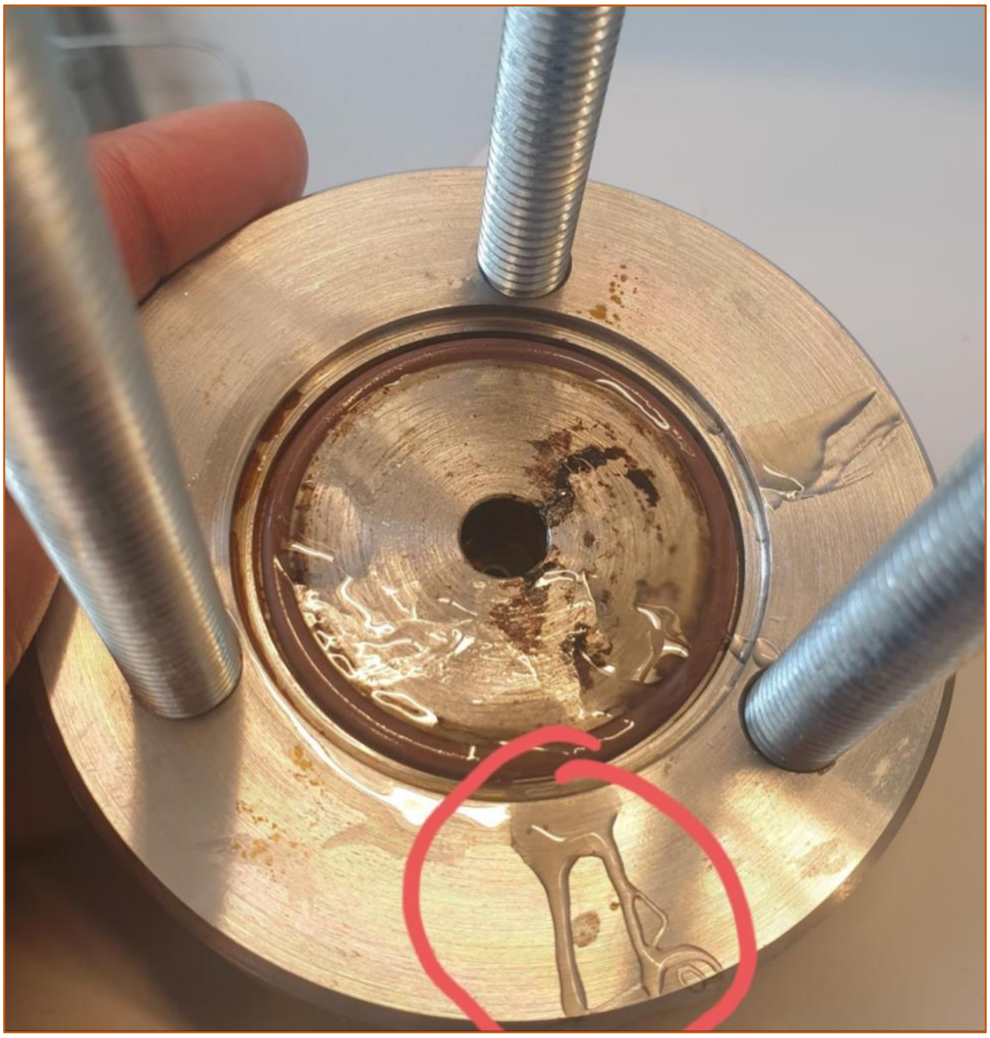

Figure 10.16 – Viscous-gel like ThermaSet produced on the outlet of sample 5

10.2.2 Squeeze ThermaSet

10.2.2.1 Sample 4 and 5

Samples 4 and 5 was tried injected with the recipe of ThermaSet optimized for squeezing. The resin did unfortunately not contain dye upon injection as it was simply forgotten. The injection for Sample 4 proved successful. Sample 5 did not inject with the resin optimized for squeezing. This was most likely due to the fact that Sample 5 was saturated with the ThermaSet optimized for plugging, having a higher viscosity.

10.2.2.2 Sample 2 and 3

Samples 2 and 3 were tried injected with the resin optimized for squeezing, containing dye. Neither of the injections rendered successful. Sample 3 did inject for some time before the flowrate went to zero. The sample experienced backpressure in the process of disconnecting the sample, resin shot out of the inlet. In Figure 10.17 the color of the dyed resin that would be injected is green. However, in Figure 10.18 the color of the residual resin in Sample 3 is altered. Bubbles are also visible within the residual resin on Sample 3.

Sample 2 did not inject; however, it has a larger permeability than Sample 3. From Figure 10.19 it appears as though the dye has colored the cement matrix. Looking at Figure 10.20, the residual resin after disconnecting the sample from the test cell is a very thick gel-like substance. The reason for this is uncertain and was not answered by the manufacturer. Sample 2 experienced a collapse of the cement core (Figure 10.8), as the viscosity of the squeeze ThermaSet is 40 times that of water, it is possible the resin has plugged the apertures and/or microannuli using the cement particles from the collapsed area and is part of the reason for the unsuccessful injection.

Figure 10.17 – Dyed resin used in treatment experiment, for color comparison.

Figure 10.18 – Sample 3 disconnected after failed injection. The color of the resin is altered and there are visible bubbles in the resin.

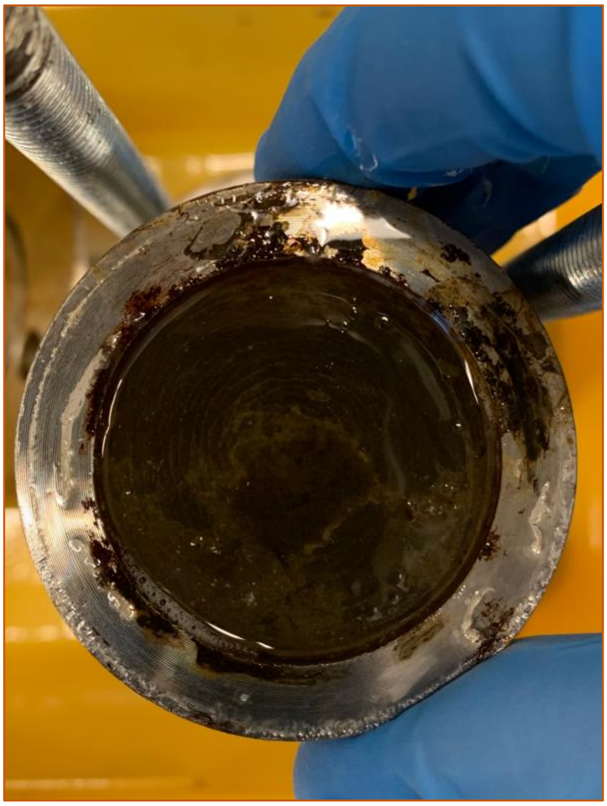

Figure 10.19 – Sample 2, the cement appears to have been colored by the dye

Figure 10.20 – Sample 2, residual resin is altered. It turned gel-like and highly viscous

10.3 Reduction in Permeability

The samples that proved successful injection of ThermaSet and Sample 5 were subject to permeability measurements post treatment. The permeabilities after treatment are listed in Table 10.3.1. From Table 10.3.2 the permeability values and hydraulic aperture are represented as a reduction in percentage.

*Table 10.3.1 – Permeability values and hydraulic aperture **after** treatment*

Sample	Fracture Permeability [μD]	Hydraulic aperture [μm]
2	NA	NA
3	NA	NA
4	0.473	0.547
5	0	0
6	164.923	3.853

Table 10.3.2 – Permeability values and hydraulic aperture reduction in percent

Sample	Fracture Permeability Reduction [%]	Hydraulic aperture Reduction [%]
2	NA	NA
3	NA	NA
4	86.486	48.683
5	100	100
6	82.608	44.181

10.3.1 Sample 4 and 6

Both Samples 4 and 6 was successfully injected with resin, from Table 10.3.2 the samples experienced a permeability reduction of 86 and 83 % respectively. The permeability of Sample 6 before treatment was based off of two measurements, however, post treatment the Sample was able to endure 4 measurements before exceeding the pumps capacity.

10.3.2 Sample 5

Sample 5 having been treated twice showed no flow during permeability testing. In fact, Sample 5 provided both negative and positive values for flowrate at higher or lower pressures (Figure 10.22). The positive and negative values are presumably caused by tolerances of the pump. Sample 5 would thus have zero water flow through the microannuli and/or any flaws post treatment.

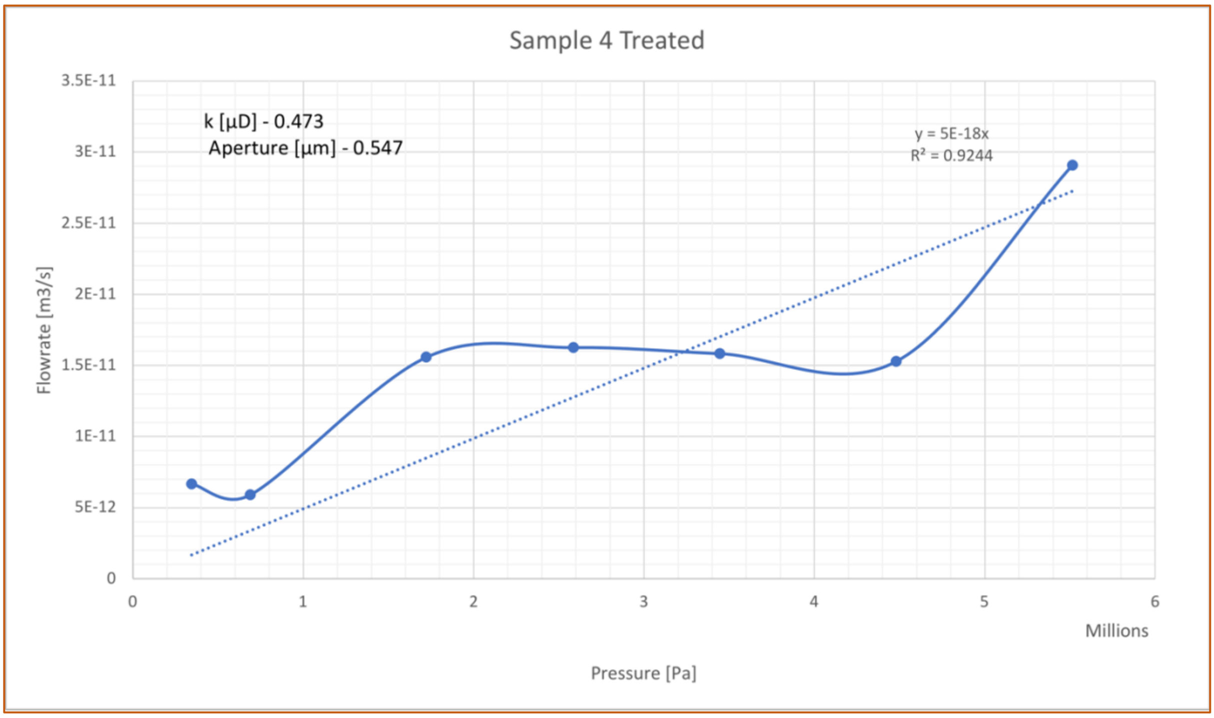

Figure 10.21 – Flow measurements of Sample 4 after treatment

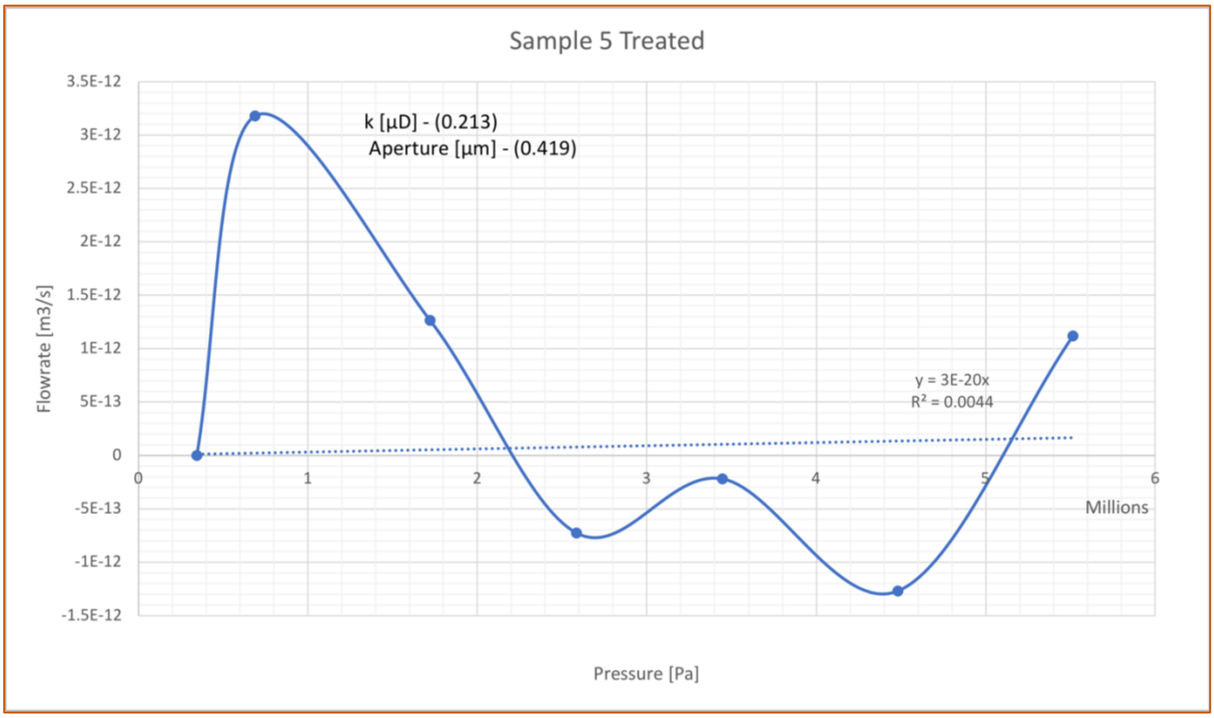

Figure 10.22 – Flow measurements of Sample 5 after treatment

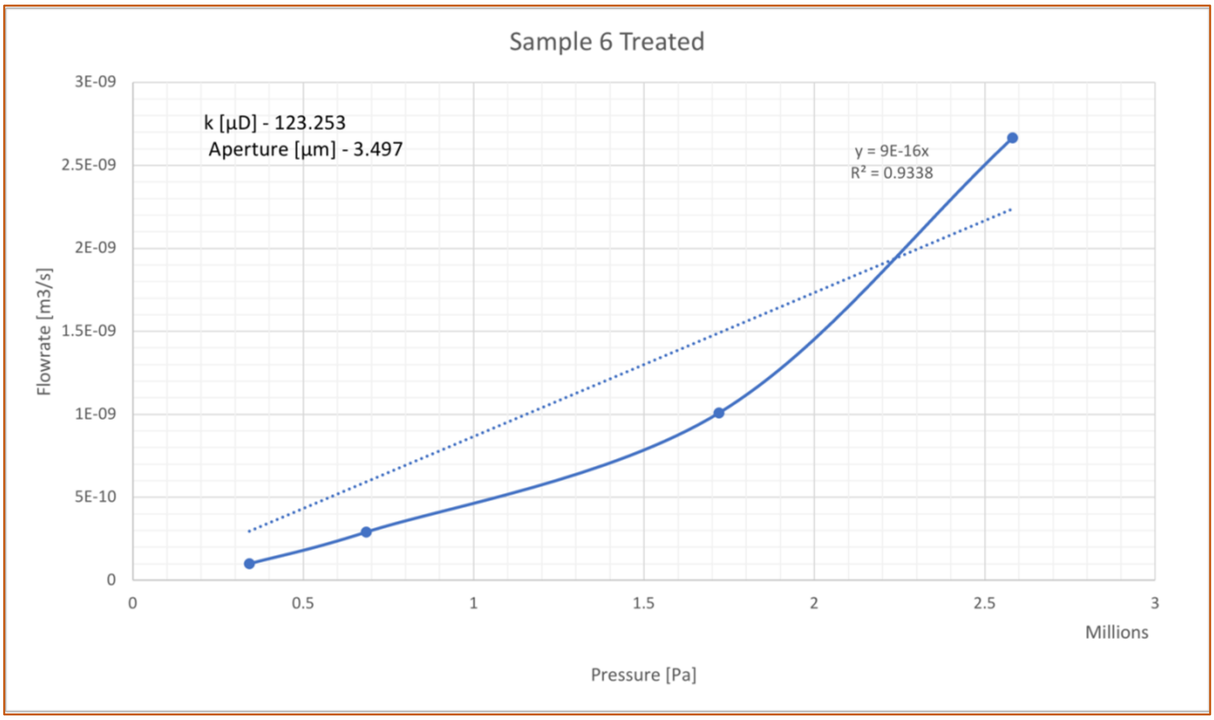

Figure 10.23 – Flow measurements of Sample 6 after treatment

11 Conclusion

Good cement typically has a water permeability in the order of 0.01 μD or less (Corina et al., 2021). The samples subjected in this thesis does all experience leakage, giving them a much higher permeability than what is considered for good cement. Stormont et al., 2018 concluded that cement hydration along with temperature and pressure cycling are of the most important mechanisms for microannuli creation. Given the experimental methodology of the samples subjected here, the occurrence of leakage is most likely due to cement hydration. The leakage could occur through microannuli; however, the leakage could also occur through any number of flaws in the cement or smaller channels at the cement-casing interface.

The treatment aspect of the thesis is somewhat inconclusive. Three out of five samples were treated, and permeability tested post treatment. Although the three samples did experience a reduction in permeability, two different materials were used and one of the samples did not contain dye. This in consideration with Sample 2, having a larger permeability than Sample 4, not injecting with the ThermaSet for squeezing containing dye and Sample 4 did inject without dye gives more questions than answers.

The treatment did indeed reduce the permeability for three samples, however, there are too many unknown factors concerning the dye and as to why a sample with a lower permeability would inject compared to a sample with a larger permeability to render a definite conclusion. Sample 5 and 2 altered the ThermaSet to a gel-like substance, the reason is unknown and was not answered by the manufacturer. Sample 6 having the largest permeability of the samples experienced a reduction in permeability of 83%. However, the sample experienced flowrates that exceeded the pumps capacity despite having an 83% reduction in permeability.

11.1 Suggested improvements for future work

In hindsight there are room for improvements. The use of dye should have been avoided as it is an unnecessary source of error considering the findings in this experiment. Should the dye be used, better routines would have ensured that all samples were injected with dyed ThermaSet. The use of two different recipes of ThermaSet should also have been avoided considering the difference in viscosity. This makes the samples incomparable, really.

In future work, the aspect of storage could be improved. How does the storage conditions affect the permeabilities of the samples? A systematic study of the underlying causes for the spread in permeabilities could also be of interest.

Another interesting extension of this thesis could compare the difference in permeabilities using gas versus water. Permeability measurements under confining pressure could also be of interest in future work.

References

ACI, A. C. I. What is autogenous shrinkage? Retrieved from https://www.concrete.org/tools/frequentlyaskedquestions.aspx?faqid=862

AER, A. E. R. (2021). *Directive 087: Well Integrity Management*. Retrieved from

Al-Ansari, A., Al-Refai, I., Al-Beshri, M., Pino, R., Leon, G., Knudsen, K., & Sanabria, A. (2015). *Thermal activated resin to avoid pressure build-up in casing-casing annulus (CCA).* Paper presented at the SPE Offshore Europe Conference and Exhibition.

Ali, W., Mata, F. J., & Al-Turki, F. A. (2021). *A Novel Approach to Successfully Cement the Long Horizontal Liners Across Khuff-C Reservoir in High-Pressure Gas Producer Land Wells: A Case History from Saudi Arabia.* Paper presented at the International Petroleum Technology Conference.

API, R. (2013). 10B-2: Recommended practice for testing well cements. *API Recommended Practice B, 10*.

Beharie, C., Francis, S., & Øvestad, K. H. (2015). *Resin: An Alternative Barrier Solution Material.* Paper presented at the SPE Bergen One Day Seminar.

Bois, A.-P.-P., Garnier, A., Rodot, F., Saint-Marc, J., & Aimard, N. (2011). How To Prevent Loss of Zonal Isolation Through a Comprehensive Analysis of Microannulus Formation. *SPE Drilling & Completion, 26*(01), 13-31. doi:10.2118/124719-pa

Boukhelifa, L., Moroni, N., James, S. G., Le Roy-Delage, S., Thiercelin, M. J., & Lemaire, G. (2004). *Evaluation of Cement Systems for Oil and Gas Well Zonal Isolation in a Full-Scale Annular Geometry.* Paper presented at the IADC/SPE Drilling Conference.

Carroll, S., Carey, J. W., Dzombak, D., Huerta, N. J., Li, L., Richard, T., . . . Zhang, L. (2016). Review: Role of chemistry, mechanics, and transport on well integrity in CO2 storage environments. *International Journal of Greenhouse Gas Control, 49*, 149-160. doi:https://doi.org/10.1016/j.ijggc.2016.01.010

Corina, A. N., Skorpa, R., Sangesland, S., & Vrålstad, T. (2021). Simulation of fluid flow through real microannuli geometries. *Journal of Petroleum Science and Engineering, 196*, 107669. doi:https://doi.org/10.1016/j.petrol.2020.107669

De Andrade, J., Sangesland, S., Todorovic, J., & Vrålstad, T. (2015). *Cement Sheath Integrity During Thermal Cycling: A Novel Approach for Experimental Tests of Cement Systems.* Paper presented at the SPE Bergen One Day Seminar.

Dow, C., & Glasser, F. P. (2003). Calcium carbonate efflorescence on Portland cement and building materials. *Cement and Concrete Research, 33*(1), 147-154. doi:https://doi.org/10.1016/S0008-8846(02)00937-7

Eklund, D., & Stille, H. (2008). Penetrability due to filtration tendency of cement-based grouts. *Tunnelling and Underground Space Technology, 23*(4), 389-398. doi:https://doi.org/10.1016/j.tust.2007.06.011

EPA, U. S. E. P. A. (2020, 09/09/2020). Understanding Global Warming Potentials. Retrieved from https://www.epa.gov/ghgemissions/understanding-global-warming-potentials

Garcia Fernandez, S., Matteo, E. N., Taha, M. R., & Stormont, J. C. (2019). Characterization of wellbore microannuli. *Journal of Natural Gas Science and Engineering, 62*, 13-25. doi:https://doi.org/10.1016/j.jngse.2018.12.003

Gasda, S. E., Bachu, S., & Celia, M. A. (2004). Spatial characterization of the location of potentially leaky wells penetrating a deep saline aquifer in a mature sedimentary basin. *Environmental Geology, 46*(6), 707-720. doi:10.1007/s00254-004-1073-5

Goodwin, K. J., & Crook, R. J. (1992). Cement Sheath Stress Failure. *SPE Drilling Engineering, 7*(04), 291-296. doi:10.2118/20453-pa

Gray, K. E., Podnos, E., & Becker, E. (2009). Finite-Element Studies of Near-Wellbore Region During Cementing Operations: Part I. *SPE Drilling & Completion, 24*(01), 127-136. doi:10.2118/106998-pa

Henkensiefken, R., Bentz, D., Nantung, T., & Weiss, J. (2009). Volume change and cracking in internally cured mixtures made with saturated lightweight aggregate under sealed and unsealed conditions. *Cement and Concrete Composites, 31*(7), 427-437. doi:https://doi.org/10.1016/j.cemconcomp.2009.04.003

Idiart, A. (2009). *Coupled analysis of degradation processes in concrete specimens at the meso-level. Chapter 3.*

Kamali, M., Khalifeh, M., Eid, E., & Saasen, A. (2021). Experimental Study of Hydraulic Sealability and Shear Bond Strength of Cementitious Barrier Materials. *Journal of Energy Resources Technology, 144*(2). doi:10.1115/1.4051269

Kamali, M., Khalifeh, M., Saasen, A., Godøy, R., & Delabroy, L. (2021). Alternative setting materials for primary cementing and zonal isolation – Laboratory evaluation of rheological and mechanical properties. *Journal of Petroleum Science and Engineering, 201*, 108455. doi:https://doi.org/10.1016/j.petrol.2021.108455

Nelson, E. B., & Guillot, D. (2006). *Well Cementing* (Second ed.): Schlumberger.

Newman, K., Wojtanowicz, A., & Gahan, B. C. (2001). Cement pulsation improves gas well cementing. *222*, 89-94.

NORSOK, S. N. (2021). Well Integrity in Drilling and Well Operations. In *NORSOK D-010, Rev. 5*.

Rocha-Valadez, T., Hasan, A. R. R., Mannan, S., & Kabir, C. S. S. (2014). Assessing Wellbore Integrity in Sustained-Casing-Pressure Annulus. *SPE Drilling & Completion, 29*(01), 131-138. doi:10.2118/169814-pa

Saponja, J. (1999). Surface Casing Vent Flow And Gas Migration Remedial Elimination-New Technique Proves Economic And Highly Successful. *Journal of Canadian Petroleum Technology, 38*(13). doi:10.2118/99-13-06

Stormont, J. C., Fernandez, S. G., Taha, M. R., & Matteo, E. N. (2018). Gas flow through cement-casing microannuli under varying stress conditions. *Geomechanics for Energy and the Environment, 13*, 1-13. doi:https://doi.org/10.1016/j.gete.2017.12.001

Torbergsen, H.-E. B., Haga, H. B., Aadnøy, B. S., Sæby, J., Johnsen, S., Rausand, M., & Lundeteigen, M. A. (2012). An Introduction to Well Integrity. In N. O. a. G. A. s. W. I. Forum, NTNU, & UiS (Eds.). Norske olje og gass.

Vignes, B., & Aadnøy, B. S. (2009). Well-Integrity Issues Offshore Norway. *SPE Production & Operations, 25*(02), 145-150. doi:10.2118/112535-pa

Vrålstad, T., Saasen, A., Fjær, E., Øia, T., Ytrehus, J. D., & Khalifeh, M. (2019). Plug & abandonment of offshore wells: Ensuring long-term well integrity and cost-efficiency. *Journal of Petroleum Science and Engineering, 173*, 478-491. doi:https://doi.org/10.1016/j.petrol.2018.10.049

Vrålstad, T., Skorpa, R., Opedal, N., & De Andrade, J. (2015). *Effect of Thermal Cycling on Cement Sheath Integrity: Realistic Experimental Tests and Simulation of Resulting Leakages.* Paper presented at the SPE Thermal Well Integrity and Design Symposium.

Wang, T., Peng, Q., Liu, W., & Feng, L. (2010). Study on Shrinkage of Concrete Based on BP Neural Network. *Advanced Materials Research, 163-167*, 3249-3257. doi:10.4028/www.scientific.net/AMR.163-167.3249

Watson, T. L., & Bachu, S. (2009). Evaluation of the Potential for Gas and CO2 Leakage Along Wellbores. *SPE Drilling & Completion, 24*(01), 115-126. doi:10.2118/106817-pa

Welltec. Sustained Casing Pressure. Retrieved from https://welltec.com/sustained-casing-pressure/

Wigston, A., Williams, J., Davies, L., & Ryan, D. (2019). *Technology Roadmap to Improve Wellbore Integrity SUMMARY REPORT*.

Wojtanowicz, A. K., Nishikawa, S., & Rong, X. (2001). Diagnosis and Remediation of Sustained Casing Pressure in Wells. . In: U.S. Department of Interior, Herndon, Virginia.